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EMAA AEROSOL GENERATOR DESIGN AND CORROSION STUDY

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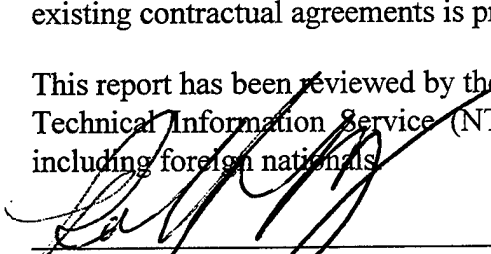TYNDALL AFB FL 32403-5319

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The design and development of an aerosol generator utilizing pyrotechnically activated EMAA is subject to two principal requirements; effective total-flooding of the protected area, and safe transfer of thermal energy derived from EMAA combustion. Because EMAA's are ineffective below a threshold airborne concentration the aerosol generator design must limit the potential for trapping excessive amounts of aerosol before it can be released into the protected area. This concern is coupled with designing a reliable system for transferring relatively large amounts of heat from EMAA combustion to both aid in the effectiveness of the aerosol suspension and prevent the possibility of secondary fires. In addition, the corrosivity potential of the various EMAA formulations on different commonly-used metals and metal alloys found in military equipment was tested and evaluated.

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aerosols; particulates; pyrotechnically generated aerosols; PGA; halon replacement; corrosion

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ABBREVIATIONS AND ACRONYMS

ASTM   American Society of Testing Methods

CFC   chlorofluorocarbons

EMAA   encapsulated micron aerosol agent

HBFC   hydrobromofluorocarbon

HCFC   hydrochlorofluorocarbon

HFC   hydrofluorocarbon

mpy   mils per year (corrosion)

PCE   pyrometric cone equivalent

PGA   pyrotechnically generated aerosol

SFE   Spectronix Fire Extinguishant

PREFACE

This report was prepared by the Fire Testing and Research Center at the University of Florida and the Infrastructure Technology Section of Wright Laboratory (WL/FIVCF), Tyndall Air Force Base, Florida. This document provides an aerosol generator design overview and preliminary test results, and overview and results of a screening study of EMAA corrosivity.

The Start Date for the project was 1 January 1995 and the End Date was 31 December 1995. The WL/FIVCF Project Officer was MAJ Robert Tetla and the University of Florida Principal Investigator was Weilin P. Chang.

EXECUTIVE SUMMARY

A.   OBJECTIVE

The objective of this program is to design, develop, and test working prototypes of aerosol generators which utilize encapsulated micron aerosol agents (EMAA) in total-flood firefighting applications. In addition, testing procedures and results of the preliminary study of the different EMAA formulation's corrosivity of various metals and metal alloys is reported.

B.   BACKGROUND

Encapsulated Micron Aerosol Agents (EMAA) offer a promising possibility as a halon replacement for several reasons: no known ozone depleting potential (ODP) or global warming potential (GDP), and the potential for less maintenance and lower long term costs for effective fire protection. The considerations for design of a delivery system must take into account the effectiveness of the aerosol in total-flooding conditions. EMAA is ineffective in fire suppression if the aerosol concentration falls below approximately 50 grams/cubic meter. This factor necessitates a generator designed to allow relatively free flow between where EMAA combustion occurs and where the aerosol exits the generator. Because this form of EMAA is pyrotechnically generated, the resulting thermal energy must be dissipated and transferred within the generator to prevent high-temperature aerosol from exiting the generator and increasing the possibility of secondary fire within the protected area. In addition, design and placement must account for free convection and accumulation of heated aerosol at the top of the protected area. Thus, the aerosol generator design must find a necessary balance between two equally important criteria: limiting restriction of the flow of exiting aerosol to prevent any excessive trapping of aerosol particles within the generator and providing a system which provides enough contact with effective thermally conductive material to cool the exiting aerosol to an acceptably safe temperature. Beyond these two primary concerns are issues of long-term maintenance, effective primary and secondary activation systems, size, weight, and potential cost that need to be further examined and evaluated to determine the overall feasibility of this type of aerosol generator as a

potential halon replacement. In addition, given the composition of the EMAA formulations, the types and rates of corrosion which may occur to various metals and metal alloys upon combustion and discharge must be examined. It is important to consider that experimental corrosion data is to be used only as an indicator, rather than extrapolated to provide anticipated long-term rates.

C. SCOPE

References found in many different journals, books, conference proceedings, patent applications, and other sources were found through extensive library searches. In addition, the USAF/CGET AEROSOL REFERENCE Database was utilized.

D. RESULTS

Testing of the evolution of various generator designs resulted in acceptable results with regard to fire suppression efficiency. Principle drawbacks are the potential for secondary fire produced from the combustion of the EMAA, dissipating the extensive heat generated by combustion, and the possibility of extreme pressures developed from combustion breaching the generator housing. Results of the corrosion study indicated SFE formulation B to be the least corrosive, and found SFE formulations A and C notably corrosive to steel (607 and 1020), aluminum, and magnesium.

E. CONCLUSIONS

Consideration must be given as to whether the relative fire-extinguishing capabilities of the pyrogenic SFE generator outweigh the possibility of secondary fire and possible generator housing breach. Refractory cooled generators were successful in lowering aerosol temperatures to safe levels. The various SFE formulations are effective enough to warrant further study of a non-pyrogenic aerosol generation and delivery system. SFE formulations appear to be suitable for exposure to Monel 400, stainless, nickel, brass, zinc, and copper.

F.   RECOMMENDATIONS

Further study of SFE formulations should be in the area of non-pyrogenic generation. Any further studies of SFE formulation corrosion should concentrate on the various steel alloys, aluminum, and magnesium.

SECTION I

INTRODUCTION

In accordance with the desires of the international scientific community, halon production in the developed world ceased at the end of December 1993, and as a result, acceptable replacements are being considered and tested. Most of the announced agents are "first-generation" agents—hydrochlorofluorocarbons (HCFC), hydrofluorocarbons (HFC), perfluorocarbons (PFC or FC), and hydrobromofluorocarbons (HBFC), whose drawbacks in terms of effectiveness, global environmental impact, or regulatory acceptance make them only short-term alternatives. As a result, candidates that are effective but have minimal global environmental impacts are being researched considerably. Of these candidates, aerosol agents are potentially promising.

Aerosol agents are one class of agent identified as potentially effective and with minimal global impact. The size of the particles in these agents is extremely small, in the range of 1 to 50 μm, which may allow the particle to remain suspended in the air for finite periods and could provide total-flood protection similar to that provided by gaseous agents, such as Halon 1301. Two technologies based on particulates have been identified—water mist and particulate aerosols, which are similar to dry agent chemicals but in smaller particle sizes. The particulate aerosols are most commonly pyrotechnically generated aerosols (PGA); however, particulate aerosols generated by non-pyrotechnic methods are being researched. Both water and dry chemicals have been used extensively in the past for firefighting applications, but the large droplet or particle sizes have prevented their use in total-flood applications, where the ability to flow around obstacles and to remain suspended in the air for extended periods is critical. Many researchers believe that the small particle sizes generated in water mists and PGAs (or other small particle techniques) may permit their use as total-flood agents.

SECTION II

AEROSOL TECHNOLOGY OVERVIEW

A. BACKGROUND

Halons have been used for many years to fight fires. However, a 1974 article by M. J. Molina and F. S. Rowland identified halogenated compounds such as halons and chlorofluorocarbons (CFC) as potentially depleting the earth's ozone layer (Reference 1). In 1978, the United States banned the use of CFC's in nonessential aerosol products. Despite this action, the global production of CFC's and halons continued and regulatory actions occurred. The Montreal Protocol, an international treaty signed in 1987, placed a cap on the consumption of halons. Effective 1 January 1994, halon consumption (production plus imports minus exports) was phased out in the United States and other developed nations.

A concentrated effort to develop substitutes for halons has been undertaken over the past few years. Halon substitutes can be divided into two types: replacements and alternatives. Replacements are halon-like agents, e.g., halocarbons. Alternatives are non-halon-like materials sometimes called "not-in-kind" agents (e.g., dry chemicals, inert gases, foam, water, carbon dioxide). Nearly all work on halon replacements has focused on halocarbons. Owing to increased concerns about global warming, atmospheric lifetime, and ozone depletion, however, halocarbons are becoming less acceptable as halon replacements, and alternatives are receiving increased attention. Recently, two alternative technologies—water misting and low-residue particulates—have come to the attention of researchers. These technologies may allow the use of water or dry chemical in reduced quantities to provide acceptable fire protection. Since the amount of agent required is reduced, secondary fire damage due to agent residue may also be significantly reduced.

B. INTRODUCTION

The search for replacements and alternatives for the halon family of chemical fire suppressants has coincided with the development of novel materials and techniques that provide new options for fire protection. One class of materials that has good potential for filling several roles

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formerly performed by halons is solid particulate fire suppression aerosols. Originating as solid materials, micron-size solid aerosol particles are generated via combustion of a solid material consisting of a combination of oxidizer, reducer, and binder. Researchers are pursuing the development of solid particulate aerosol fire suppressants for their potential both as an alternate to Halon 1301 fire protection systems and as a fire protection method of choice for certain applications. Researchers are also investigating a wide range of liquid aerosols, such as water and halocarbon mists, both of which have demonstrated significant fire suppression effectiveness for relatively small quantities of originating material.

Aerosol science or particle mechanics draws from several scientific disciplines to formulate the science that underlies its principal areas of research. Understanding the thermodynamic interaction of aerosols with fire propagation mechanisms is a new subset of aerosol science that has the potential for creating a wide variety of fire suppression options.

C.   AEROSOL CONCEPTS

Aerosol refers to a system of liquid or solid particles suspended in a gaseous medium. Aerosols are generally defined as stable or quasi-stable systems with the bulk of particles being < 1 μm in diameter. Note, however, that water mists are often designated as aerosols, and such mists are usually made up of water droplets with diameters of 50 μm or more. Aerosols affect visibility, causing some degree of obscuration, especially in the size range of 0.1 to 1 μm. The collective term "particulate" is commonly used to refer to both solid and liquid (particle and droplet) components of an aerosol when differentiation of phases is unimportant. Here, however, the term "particulate" is used to refer to solid aerosols. Several common aerosols are fumes, smoke, mists, fog, and haze.

Fumes resulting from chemical reactions may become aerosols via agglomeration of molecules due to high Brownian diffusion rates. Particle sizes vary greatly as a function of temperature and gas volume. Once formed, separation and rediffusion become very difficult. Metal fumes have particle sizes on the order of 0.5 μm. Smoke is an aerosol resulting from combustion of fuels. Like fumes, smoke has particle sizes on the order of 0.5 μm.

A solid aerosol particle can have a wide variety of shapes, but is often considered to be virtually spherical for analysis purposes. The radius, r, or the diameter, d_p, can therefore have several definitions. Because most studies utilize the projected image of the particles, the dimension of the particle is related to the analysis technique. The Feret diameter is the maximum edge-to-edge distance of the particle, while the Martin particle diameter is the length of a line that separates the particle into two portions of equal area. The aerodynamic diameter, d_{ae}, is the diameter of the spherical particle of unit density that would exhibit the same aerodynamic properties as the aerosol particle. The Stokes' diameter, d_{St}, is the diameter of a sphere that would have the same density as the aerosol particle (Reference 2).

Based on the state of the suspended substance, liquid or solid, dispersion and condensation aerosols are differentiated. Dispersion aerosols are formed by the atomization of solids and liquids, while condensation aerosols are formed via the condensation of superheated vapors or chemical reactions in the gaseous phase. In general, dispersion aerosols are coarser than condensation aerosols.

D.   AEROSOL DYNAMICS

The dynamics of aerosols is an important consideration for two reasons. First, the ability of the particles to remain suspended is obviously connected to the particle size and the residence time of the fire suppressant. Second, if aerosols are to replace gases in certain applications, they must be able to flow around obstacles.

The suspension time of an aerosol is governed by Stokes' Law, which predicts the terminal velocity of the particle through air and consequently the residence time of the aerosol. As particle size increases, the inertial and viscous forces of the fluid come into play. For larger particle sizes, the Stokes' Law predictions must be recalibrated for viscous drag forces.

The ability of the fire suppressant aerosol to flow around obstacles is required for it to be able to penetrate around and behind objects and into small spaces. The larger the particle size, the less able the particle will be to change direction, causing it to impinge on the obstacle. This property is called impaction and is governed by Stokes' number or the impaction parameter, which is the

dimensionless ratio of the particle stopping distance to the characteristic dimension of the obstacle or flow geometry (Reference 3).

Dispersion of an aerosol fire suppressant is an important consideration in evaluating effectiveness. The dispersion characteristics of the aerosol are also a function of the aerosol particle size. In general aerosol particles vary widely in size, from 1 nm to about 1 mm as the upper limit. Coarse particles with r >1 μm have a dispersion rate that is a function of diameter. Particles in the range, 0.1 μm < r < 1.0 μm, have transition properties. Very fine particle aerosols with r < 0.1 μm are dispersed proportional to r^2 and the particle velocity, v.

The loss of aerosol particles in suspension can be attributed to several phenomena: sedimentation, diffusion, and coagulation. Again, the size and velocity of the aerosol particles are the driving force. Larger particles, r $\geq$ 1 μm, will tend to fall and be lost via sedimentation. Smaller, submicron particles, will tend to diffuse out to the walls of containment via Brownian motion. Coagulation, the formation of larger particles from smaller particle via collisions, is caused by thermal, electrical, molecular, hydrodynamic, and several other forces.

E.   FIRE SUPPRESSION AEROSOLS

A solid particulate fire suppression aerosol is a dispersion aerosol that is delivered to the protected space. Aerosols have been generated by combustion of a solid tablet. Prior to the development of the particulate aerosols, dispersion aerosols were created via crushing, grinding, blasting, or drilling of solid matter. The particle size reduction is directly related to the energy expended on crushing or grinding and other factors such as the brittle or plastic nature of the material, the porosity of the solid, and the presence of crystal flaws and sites of weakness. Physico-chemical reactions using condensation processes have also been used to generate solid particulate aerosols. Salts fused on heating wires have been used to generate aerosols via incandescence in inert gas atmospheres, the temperature being a function of the energy required to produce nuclei.

Solid particulate fire suppression aerosol particles are on the order of 1-3 μm in diameter. At 1 atmosphere, these particles will have a terminal velocity of about 10^{-4} cm/s according to Stokes'

Law. Diffusion losses are also predicted to be very small. The result is that these particles will remain suspended in the protected space for times on the order of tens of minutes to several hours.

Pyrotechnically generated solid particulate fire suppression aerosols are initially a solid material that can originate in a variety of forms: solid, powder, or gel. The active components (an oxidizer and a reducer) are combined with a filler. These components are ground into a fine powder and mixed with an epoxy resin binder. Upon ignition of the material, the combustion products are ejected as a dispersion aerosol, with the solid particles floating in the air with the gaseous components.

The products of combustion of most fire suppression PGAs are 40 percent solid particles and 60 percent gaseous products. The gaseous products consist of N_2, CO_2, CO, H_2O, O_2 and traces of hydrocarbons. The solid particles are various solid salts, depending on the formulation of the originating solid.

F.   EXTINGUISHMENT MECHANISMS

Successful fire suppression requires that one or more of the four factors that tend to propagate a fire be interrupted. These factors and their associated suppression mechanisms are shown with the action of the aerosol as a fire suppressant (Table 1). Solid particulate aerosols, like dry chemicals, are hypothesized to function via several mechanisms to suppress fires.

TABLE 1. FACTORS GOVERNING FIRE PROPAGATION.

Factor	Suppression Mechanism	Aerosol Actions
Fuel	Removal	N/A
Oxygen	Exclusion	Inert gas formation
Heat	Absorption	Cooling via decomposition/vaporization
Chain reaction	Inhibition	Absorb active species

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Chemical inhibition of the chain reaction is hypothesized to occur via catalytic combination of the active free-radical species. There is also significant evidence that heat absorption and cooling via decomposition and vaporization of the solid particles is an important mechanism for flame extinguishment. The final mechanism may be oxygen dilution in the flame region as the chemical reaction of the particles and active species produces inert gases such as CO_2, causing localized low oxygen conditions.

1.   Chemical Inhibition Interactions

Chemical inhibition is a function of several variables. Depending on the temperature at the point of interaction, the aerosol particles can act by homogeneous inhibition, as shown in the following examples for a potassium-containing agent:

$$K + OH + M \longrightarrow KOH + M \tag{1}$$

$$KOH + H \longrightarrow H_2O + K \tag{2}$$

$$KOH + OH \longrightarrow H_2O + KO \tag{3}$$

where M is third-body molecule (e.g., nitrogen or argon), and H and OH are active species. The extinguishing process is in fact similar to that of the halons.

Chemical precursors that interact with the active species are often the alkali metals K, Na, Cs, Rb, Sr and associated anions such as CO_3, HCO_3, SO_4, NH_4, and PO_4. The alkali-metal salts have been shown to be especially effective fire suppressants. The potassium salts are generally superior to the sodium salts and the anion associated with each is an important factor in fire suppression effectiveness (Reference 4). For example, alkali metal oxalates are particularly effective compared to bicarbonates.

Fragmentation of dry chemical agents, such as alkali metal salts, increases the particulate-specific area for interaction. Large dry chemical particles may decompose in flames to produce inhibiting species, such as alkali hydroxides. To allow decomposition to occur, residence time in the flame is important. For large particles, the appropriate residence time may be difficult to achieve because the mass of the particle will cause it to fall rapidly through the flame. In the

case of 1 μm aerosol particles, the residence time required to produce the reactive species is far shorter and the diffusion property of the small solid particle will tend to maintain its availability in the flame. The combination of these effects may result in the increased effectiveness of particulate aerosols compared to dry chemical fire extinguishants of similar composition. Clearly, the penetration of the flame by these particles is a complex phenomenon and must include considerations of density, momentum, and the convection characteristics of the scenario in addition to the particle size. The large particle sizes of dry chemicals may have some advantage in flame penetration compared to the small aerosol particles because of their momentum.

2.   Thermal Cooling Mechanisms

Relatively recent evidence suggests that much of the effectiveness of dry chemicals can be attributed to thermal and heat extraction mechanisms such as heat capacity, fusion, vaporization, and decomposition (Reference 5). At certain particle sizes, depending on the dry chemical powder composition, a sizable increase in extinguishing effectiveness is achieved that can be explained by flame heat removal (References 6 and 7). This occurs at limit temperatures that are a function of the flame and extinguishant properties. The particle size at which the step increase in effectiveness occurs is the limit size, S_L, defined as the largest particle size that completely reacts with the flame. The S_L varies with the composition of the dry chemical constituent of the formulation. Above S_L, heat extraction is due to the heat capacity of the solid particle alone. Below S_L, several mechanisms are effective including heat capacity, dissociation, decomposition, and vaporization. Plots for five dry chemicals—$KHCO_3$, Monnex, $NH_4H_2PO_4$, $NaHCO_3$, and KCl—are contained in Reference 6. These graphs provide valuable insights into the behavior of dry chemicals as a function of particle diameter as well as impetus to examine dry chemical aerosols that appear to be especially effective.

In addition to the differences in relative effectiveness of various dry chemical formulations, for the same alkali metal the fire suppression efficiency as a function of the anion appears to be as follows:

Oxide > cyanate > carbonate > iodide > bromide > chloride > sulfate > phosphate

The generation of alkali hydroxide in the flame is believed to be the reason for the relative effectiveness of the various anions.

G.   APPLICATIONS

1.   Pyrotechnically Generated Aerosols

The aerosol generated when an PGA tablet is ignited has several properties that differentiate it from both gaseous agents and dry chemicals. In fact, PGA could be said to be an intermediate agent between these two extremes in fire suppression techniques. The following are several of the key characteristics and features of PGA that influence the design of applications:

a. Similar to (but less effectively than) a gaseous agent, PGA can flow around barriers and obstacles, behaving like a gas in its basic transport properties. It can be introduced into ductwork and be delivered to an area via forced convection. Dry chemicals, in contrast, are more limited by obstructions.

b. PGA has excellent Class B fire suppression characteristics, similar to those of dry chemicals. Both PGAs and dry chemical agents are about 4 times as effective as Halon 1301 per unit mass and up to 10 times as effective as the proposed first-generation replacements for Halon 1301.

c. PGA initiation is independent of oxygen supply and can, therefore, be effective under or within a liquid or at altitudes where oxygen concentrations are low.

d. Initiation of PGA can be active via electrical ignition or passive via self-ignition due to interaction with a fire.

e. The delivery rate of PGA is a function of its composition, form (solid, powder, gel), and the delivery system. The aerosol is generated by combustion of the PGA material; variations in the active component, oxidizer, and reducer dramatically affect the burn rate, perhaps up to a difference of two orders of magnitude.

f. PGA does not require piping, pressure cylinders, or valves. A device for containing the PGA solid material is all that is normally required. Pressure testing, weighing, pressure/leak detection, and other maintenance and testing of cylinders/pipes/nozzles/valves are not required.

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The low weight to extinguishment capability of PGA provides tremendous performance advantages for weight- and space-critical applications. A CO_2 cylinder weighing more than 150 Kg could be replaced with about 4 Kg of PGA.

It has been demonstrated that small particle dry chemicals (below S_L in size) can be created by mechanical means. However, practical utilization of mechanically created, small diameter dry chemical compounds is limited because it is difficult to store dry chemicals for extended periods of time without compaction. Humidity also has a detrimental effect on dry chemicals and results in deterioration. The production of dry chemical solid particulate aerosols by combustion avoids these difficulties, and the solid material has an estimated 15-year shelf life. Packaging can be readily designed that provides protection even in fairly extreme environments.

This excellent performance capability and its add-on ability will enable PGA use in applications such as trucks and cars, boats and ships, engine compartment protection, fuel tanks, and numerous other applications. Where portability, expandability, simplicity, ruggedness, and cost are factors, a solid particulate aerosol system should be considered.

The major unknowns for PGAs at present are materials compatibility, especially corrosion, and application against deep-seated fires. Testing to assess aerosol performance in both of these areas is ongoing.

2.   Conclusions

The development of aerosol fire suppression systems is a newly emerging discipline that holds great promise in offering an excellent option for consideration for several fire protection roles. The delivery systems must be able to accommodate the high combustion temperature, flame generation, and potential corrosiveness of the pyrotechnically generated aerosol.

SECTION III

AEROSOL GENERATOR DESIGN OVERVIEW

A. INTRODUCTION

The design and configuration of the EMAA aerosol generator needs to take into account efficient delivery of the aerosol, and effective heat transfer to prevent secondary fire. In addition, concerns with size, weight, reusability, and cost need to be addressed. The design of the prototype incorporates a simple delivery system, machined with non-exotic materials. The potential for reusability is very good. The size and weight of the system may cause some application limitations, but reductions in both factors may be possible in refined designs.

B. WATER COOLED DESIGN OVERVIEW

As stated earlier the design of an adequately cooled delivery system involved the use of heat absorbing materials or fluids. Several trials using water as a coolant showed no immediate improvement in the exhaust temperature. Figure 1 and 2 show the design of a device with a water cooled jacket. In this design, the aerosol stream impinges on a U-tube design heat exchanger which is surrounded by water. Subsequent designs utilized four parallel delivery tubes (Figures 3 and 4) and eight delivery tubes (Figures 5 and 6) to increase heat exchange via increased water flow and contact with convective surfaces.

C. REFRACTORY DESIGN OVERVIEW

The EMAA aerosol generator design has three major components; the housing (including combustion chamber), refractory/insulation material, and ignition system. Given the problems concerning efficient thermal transfer and containment, many different coolant systems were considered. Utilizing water as the sole source of coolant is ineffective given the high combustion

Figure 1   Aerosol Generator Design, Water-Cooled U-Tube Delivery System

SWITCH
BATTERY COVER
WIRE TO S.F.E.
REMOVABLE METAL
SCREW CAP

9 VOLT BATTERY

PLATE - Z

S.F.E MATERIAL BY OTHERS
3" DIA. SCH. 40 PIPE
PLATE - A

1 1/8"

PLATE - B

3/4"

1/8" TYP.

3/8" COPPER PIPE

13"

4" SCH. 40 PIPE (STEEL)

12"

1/4" REMOVEABLE SCREW

6 3/8"

FILLET WELD PERIMETER
OF EACH PLATE B-E (TYP)

PLATE - C

PLATE - D

3/4"

3"
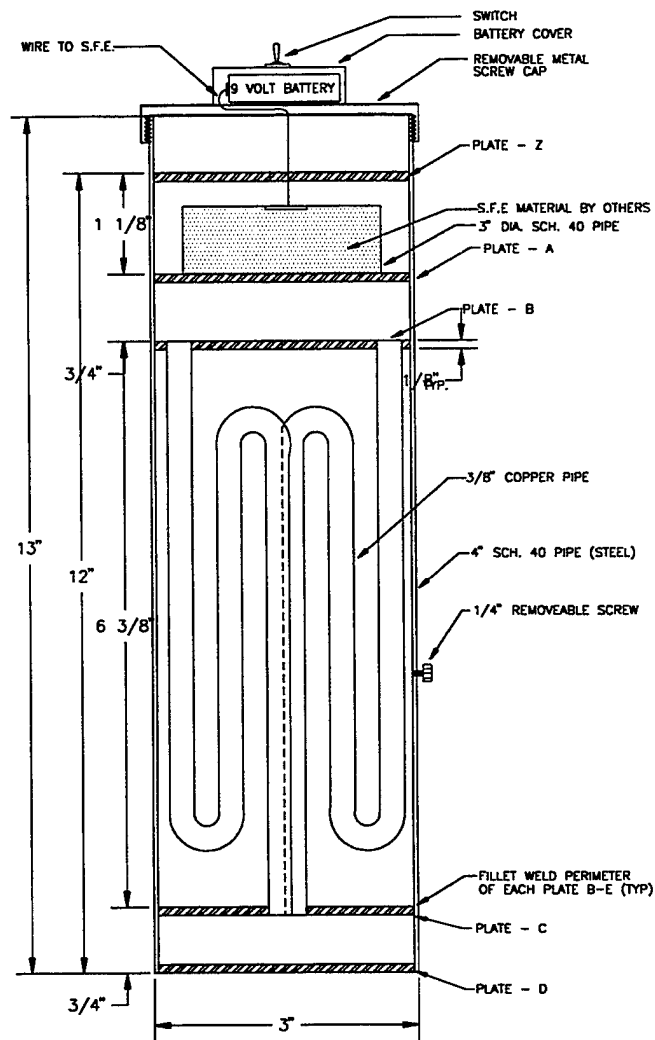
PLAN VIEW / SECTION
SCALE: 1" = 1"

Figure 2   Generator, enhanced view

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Figure 3   Water-Cooled, Four-Tube Delivery System

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WIRE TO S.F.E.
SWITCH
BATTERY COVER
(18 GAUGE SHEET METAL)
REMOVABLE METAL
SCREW CAP
9 VOLT BATTERY
PLATE — A
2 1/4"
S.F.E MATERIAL BY OTHERS
3" DIA. SCH. 40 PIPE
1 1/8"
PLATE — B
PLATE — C
3/4"
1/8" TYP.
13"
12"
3/4" DIA. PIPE TYP. OF 5
4" SCH. 40 PIPE (STEEL)
1/4" BOLT
6 3/8"
FILLET WELD PERIMETER
OF EACH PLATE & TUBE
PLATE — D
1/8" STEEL SUPPORT RING
PLATE — E
3/4"
3"
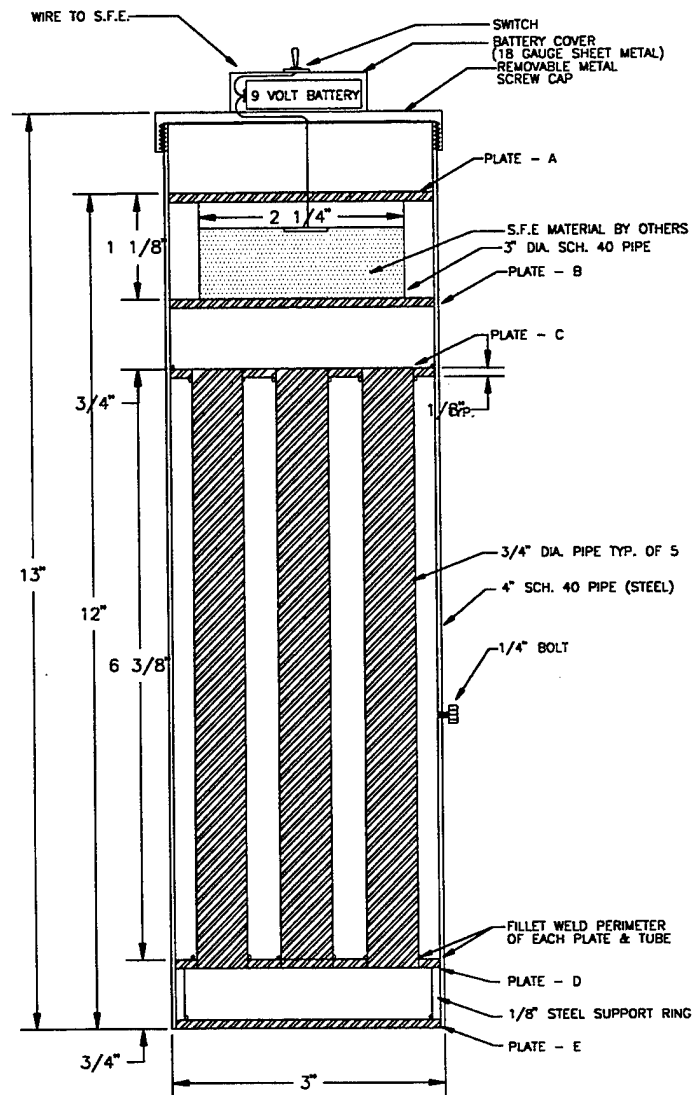
PLAN VIEW / SECTION
SCALE: 1" = 1"

Figure 4   Generator, Enhanced View

15
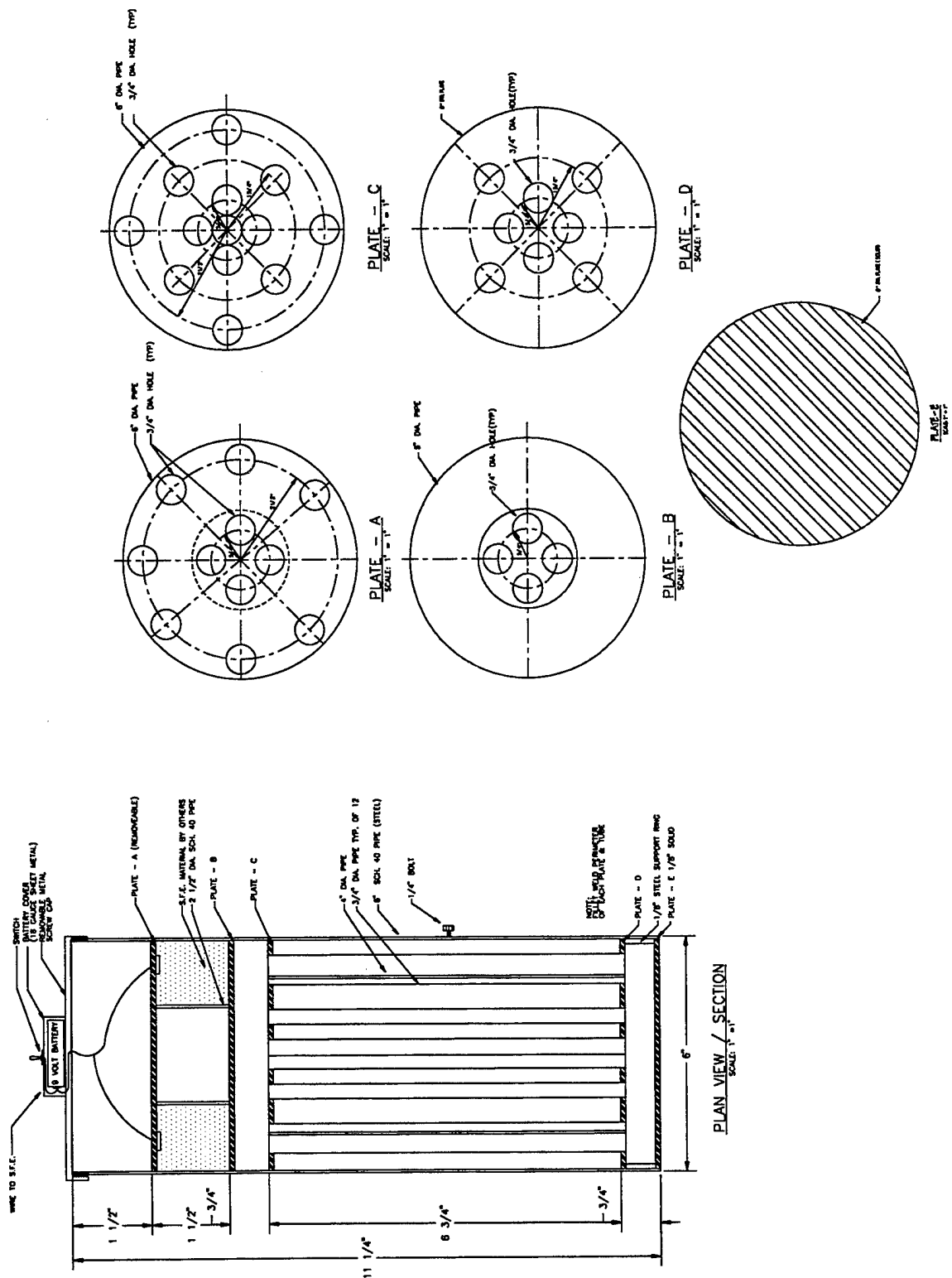
Figure 5   Water-Cooled Design, Eight-Tube Delivery System

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WIRE TO S.F.E.

SWITCH
BATTERY COVER
(18 GAUGE SHEET METAL)
REMOVABLE METAL
SCREW CAP

9 VOLT BATTERY

1 5/16"

PLATE - A (REMOVEABLE)

1 5/16"

S.F.E. MATERIAL BY OTHERS
2 1/2" DIA. SCH. 40 PIPE

5/8"

PLATE - B

PLATE - C

9 3/4"

4" DIA. PIPE
3/4" DIA. PIPE TYP. OF 12
6" SCH. 40 PIPE (STEEL)

1/4" BOLT

5 7/8"

NOTE:
FILLET WELD PERIMETER
OF EACH PLATE & TUBE

5/8"

PLATE - D
1/8" STEEL SUPPORT RING
PLATE - E 1/8" SOLID

6"

PLAN VIEW / SECTION
SCALE: 1" =1"

Figure 6   Generator Enhanced View

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temperatures of the different EMAA formulations. Aerosol cooling must be coupled with insulating the housing from the heat generated, therefore the possibility of a shapable refractory material coupled with a packed-bed of spherical refractory med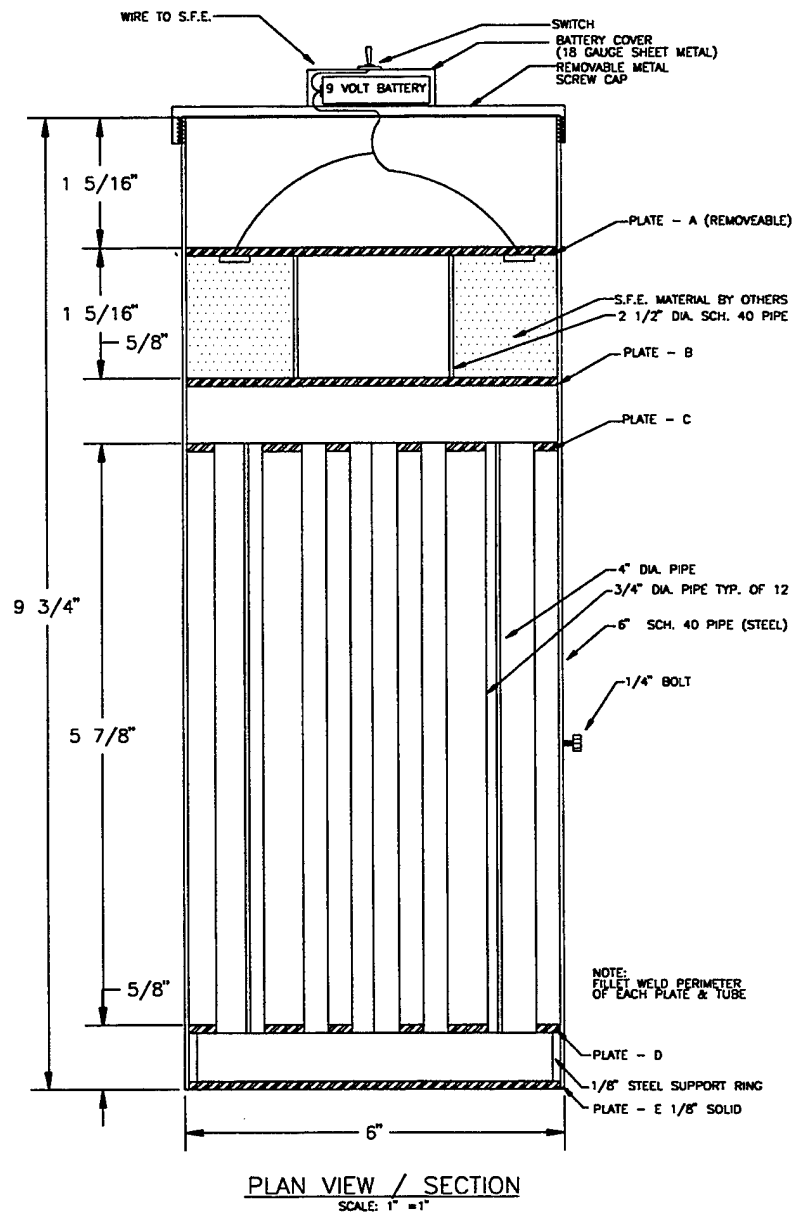ia evolved as the most feasible approach. Housing for the generator must allow for high resistance to heat and thermal shock, no pressure buildup of aerosol, maximum surface area for contact with the aerosol without trapping excess aerosol particulate matter which could limit effectiveness of fire suppression. Finally, a dependable ignition system with a fail-safe device is needed. Figures 7 and 8 show the design of the refractory-cooled generators used in the testing. Figure 9 shows the family of refractory generators developed under this program.

D. REFRACTORIES

Refractories are materials that resist the action of hot environments by containing heat energy. The type of refractories that are used in any particular application depends upon the critical requirements of the process. For example, processes that demand resistance to gaseous or liquid corrosion require low porosity, high physical strength, and abrasion resistance. Conditions that demand low thermal conductivity may require entirely different refractories. Combinations of several refractories are generally employed. There is no well established line of demarcation between those materials that are and those that are not refractory although the ability to withstand temperatures above 1100 ^{0} C without softening has been cited as a practical requirement of industrial refractory materials.

Insulating Refractories. Refractory fiber made from molten oxides may be formed into bulk fiber, blankets, boards, or blocks. Such fibers are used as back-up thermal insulation and low heat-capacity linings in kilns and reheat furnaces. These materials are used to line the interior wall of the prototype aerosol generator, and to provide an insulating interface between the combustion chamber and the top plate of the generator.
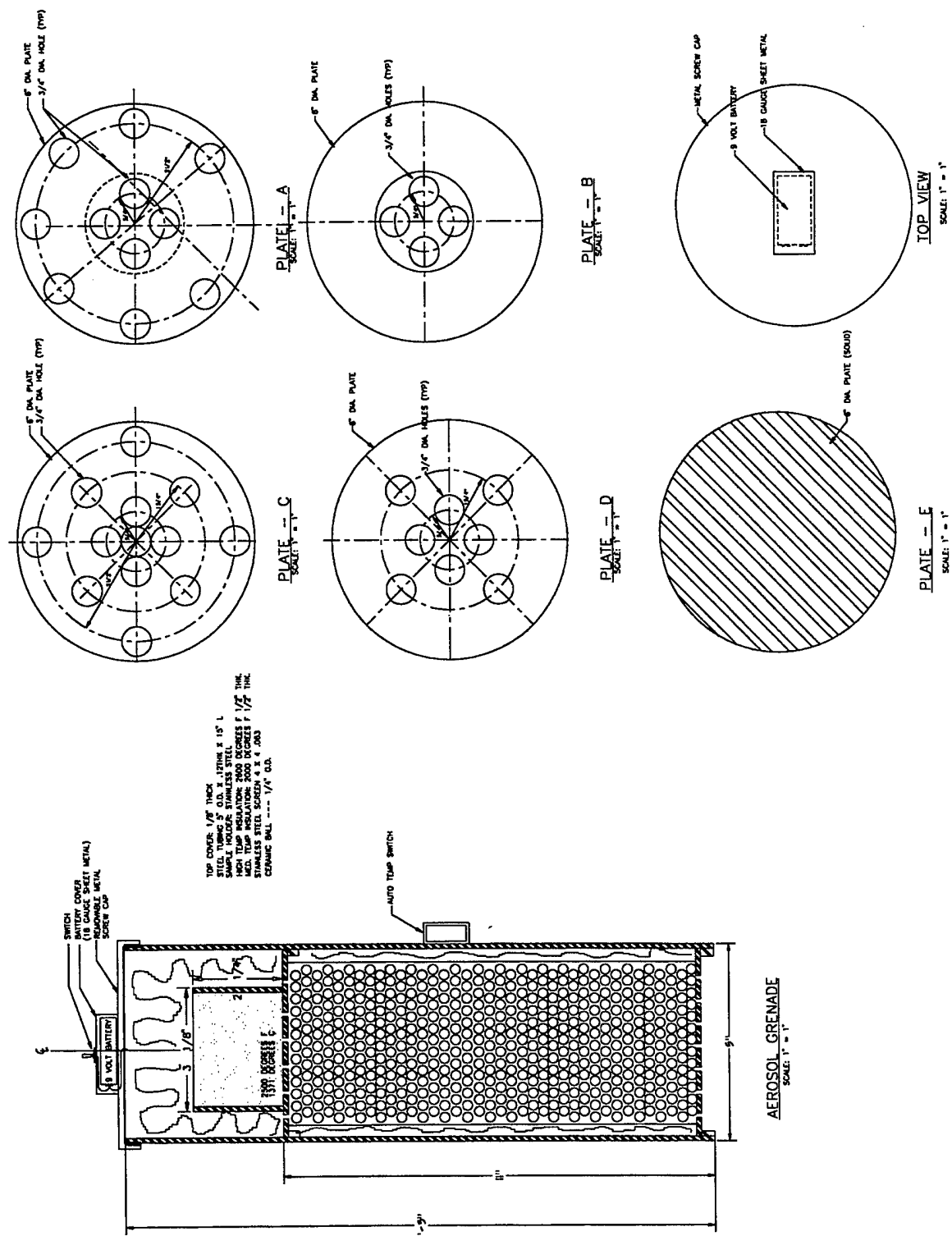
Figure 7   Refractory-Cooled Delivery System, Alumina Sphere

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SWITCH
BATTERY COVER
(18 GAUGE SHEET METAL)
REMOVABLE METAL
SCREW CAP

9 VOLT BATTERY

3 1/8"

2 1/4"

2500 DEGREES F
1371 DEGREES C

TOP COVER: 1/8" THICK
STEEL TUBING 5" O.D. X .12THK X 15" L
SAMPLE HOLDER: STAINLESS STEEL
HIGH TEMP INSULATION: 2600 DEGREES F 1/2" THK.
MED. TEMP INSULATION: 2000 DEGREES F 1/2" THK.
STAINLESS STEEL SCREEN 4 X 4 .063
CERAMIC BALL --- 1/4" O.D.

AUTO TEMP SWITCH

1'-3"

11"

5"

AEROSOL GRENADE
SCALE: 1" = 1"

Figure 8   Generator, Enhanced View

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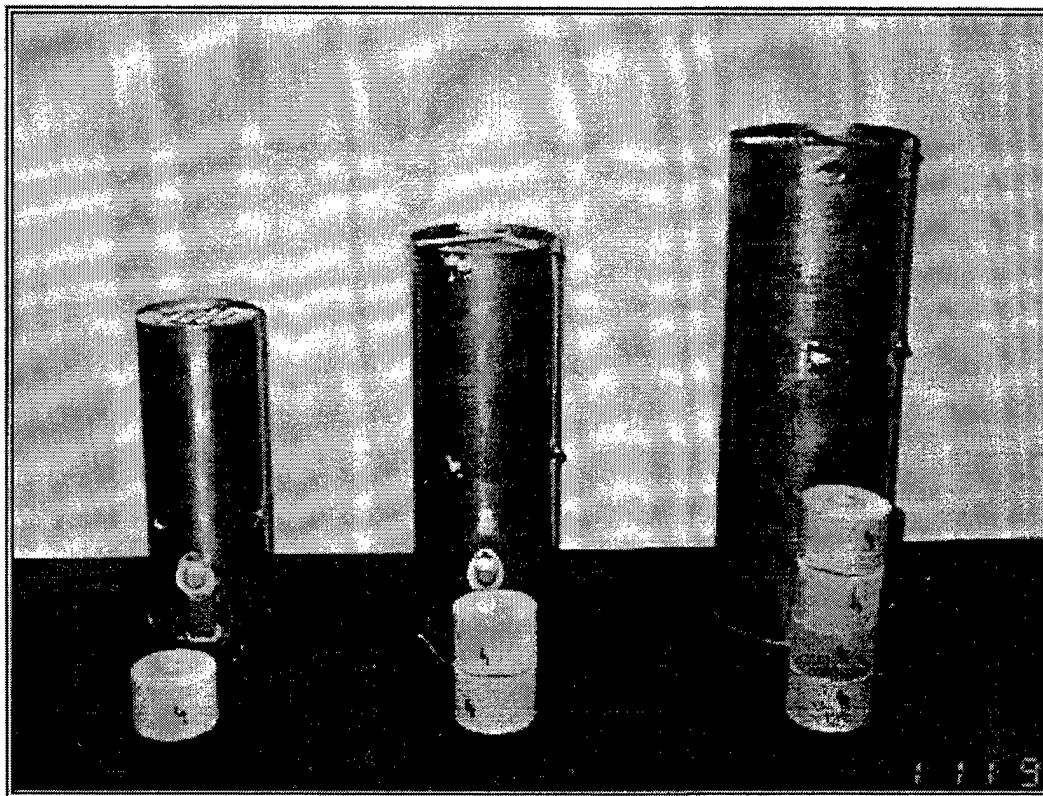
Figure 9   Refractory-Cooled Generators

Refractory Coatings. Refractory coatings are applied either by painting or by spraying to a fine-grained refractory mix at room temperature. By heating, a dense sintered coating is formed. Other techniques include flame or plasma spraying. In the former, the powdered coating or material is fed into a burner and sprayed at elevated temperatures. The pyroplastic grains form a dense monolithic coating when they impinge on the substrate. Plasma spraying is carried out in essentially the same manner, except that an electrically ionized gas plasma heats the coating powder to temperatures up to 16,700 ^{0} C. The possibility of applying a sintered refractory coating to the interior wall of the aerosol generator could be further considered for potential added efficiency of thermal transfer.

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E. COMPARISONS OF REFRACTORIES

The choice of refractories must be evaluated by using various criteria which differ depending upon chemical composition, manufacture, and size of the media. Depending upon the application, the following criteria need to be utilized.

Physical Properties. Coarse, medium, and fine-sized material contribute to the degree of packing. Usually the highest density possible is desired. Upon firing, the grains and matrix form glassy, direct, or solid-state ceramic bonds. Sintering is generally accompanied by shrinkage. Unless new components are formed that may cause expansion. Differential volume change between the coarse and fine fraction caused by differential sintering rates and formation of addition phases may create stresses. Particle size distribution, forming method, and firing process contribute to texture, whereas permeability is related to porosity, which in turn is dependent upon texture.

Mechanical Properties. The physical properties of a particular refractory product depend upon its constituents and manner in which they were assembled. The physical properties may be varied to suit specific applications. For example, for thermal insulation, highly porous product is employed, while dense products are used for slagging or abrasive conditions. The strength (modulus of rupture) at room temperature is determined by the stronger than coarse-grained degree of bonding. Fine-grained refractories generally are used for many applications and those with a low porosity are stronger than those of high porosity. However, the room temperature strength of a refractory is not necessarily indicative of the strength at high temperature because the bond strength may be due to a glassy phase that softens upon heating. Generally, high temperature strength is lower than room temperature strength. The former is a measure of the degree of solid-state bonding between refractory grains, whereas high temperature creep indicates the amount of associated liquid or glassy phases and their viscosity. The development of solid-state or direct-bonded basic brick requires high-firing temperatures, and is impeded by glassy phases. By referring to phase diagrams, refractory compositions may be designed that avoid the development of such phases.

Thermal Properties. Refractories, like most other solids, expand upon heating, but much less than most metals. The degree of expansion depends upon the chemical composition. Thermal conductivity depends on the chemical composition of the material and increases with decreasing porosity.

Specific Heat. In some applications, refractories are used for heat-exchange purposes on the regenerative principle, for instance, in blast-furnace stoves. High heat capacity is required in such applications.

Thermal Spalling. Refractories are brittle and stresses caused by sudden variations in temperatures can cause cracking and destruction. The susceptibility to thermal cracking and spalling depends upon certain characteristics of the raw material and the macrostructure of the particular refractory. Fireclay and high alumina refractories usually have a higher resistance to thermal shock than periclase refractories. Dense strong bodies withstand high stress and transmit it over large volumes; when failure occurs it is serious. Weak porous bodies, however, tend to crack before catastrophically large stresses are generated and thus are much less seriously damaged and generally remain intact.

Refractoriness. Most refractories are mixtures of different oxides, sometimes with significant quantities of impurities. Thus, they do not have sharp melting points but a softening range. Refractoriness is the resistance to physical deformation under the influence of temperature. It is determined by the pyrometric cone equivalent (PCE) test. The PCE test (ASTM C 24) measures the softening temperature of refractory materials. Inclined trigonal pyramids (cones) are formed from finely ground material, set on a base, and heated at a specific rate. The time and temperature (heat treatment) required to cause the cone to bend over and touch the base is compared to that for standard cones. The standard ASTM PCE test is relative and used extensively only for alumina-silica refractories. However, the upper service limit is generally several hundred degrees below the nominal PCE temperature since some load is generally applied to the refractory during service. In addition, chemical reactions may occur that alter the composition of the hot face and, therefore, the softening point. The relationship between PCE numbers and temperature is described in ASTM C 24.

Health and Safety Factors

Because industrial refractories are by their very nature stable materials, they usually do not constitute a physiological hazard. By far the most common industrial refractories are those composed of single or mixed oxides of Al, Ca, Cr, Mg, Si, and Zr. These oxides exhibit relatively high degrees or stability under both reducing and oxidizing conditions. Carbon, graphite, and silicon carbide have been used both alone and in combination with the oxides. Refractories made from the above materials are used in ton-lot quantities in industrial applications. Other refractory oxides, nitrides, borides, and silicates are used in relatively small quantities for specialty applications in the nuclear, electronic, and aerospace industries.

Reactions between Refractories and Gases. Reactions with gases can be quite destructive as the gases generally penetrate the pores of the refractory and destroy its structure. The refractory may either expand and crack because of the formation of new, low density compounds, or its refractoriness may be drastically reduced because of the formation of low melting compounds.

F. GENERAL PROPERTIES OF OXIDES AND ALUMINA

Oxides. At present alumina, Al_2O_3 is the most widely used simple oxide; it has moderate thermal-shock resistance, good stability over a wide variety of atmospheres, and is a good electric insulator at high temperatures. The strength of ceramics is influenced by minor impurities and microstructural features. In general, polycrystalline alumina has reasonably good and nearly constant strength up to about 1000-1100 ^{0} C; at higher temperatures, the strength drops to much less than one half of the room temperature value over a 400 ^{0} C temperature increment. Single-crystal alumina is stronger than polycrystalline Al_2O_3 and actually increases in strength between 1000 ^{0} C and 1100 ^{0} C. Fused silica glass has excellent thermal-shock properties, but devitrifies on long heating above 1100 ^{0} C and loses much of its shock resistance.

Beryllium and magnesium oxides are stable to very high temperatures in oxidizing environments. MgO has strong potential for use as a refractory material, and initial tests utilizing the first refractory-cooled generator (Figure 2) showed acceptable results with regard to heat-exchange,

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but high levels of aerosol deposition due to irregular shapes of the material led to reduced fire-suppression capabilities. MgO should be further considered for combination with other refractory materials. Above 1700 ^{0} C MgO is highly volatile under reducing conditions and in vacuum, whereas BeO exhibits better resistance to volatilization but is readily volatilized by water vapor above 1650 ^{0} C. Beryllia has good electrical insulating properties and high thermal conductivities; however, its high toxicity restricts its use. Calcium oxide, and to a lesser extent uranium oxide, hydrate readily; UO_2 can be oxidized to lower melting U_3O_8- Zirconia in pure form is rarely used in ceramic bodies; however, stabilized or partially stabilized cubic ZrO_2 is currently the most useful simple oxide for operations above 1900 ^{0} C, but probably not cost effective for consideration. Thorium oxide exhibits good properties for high temperature operation but is very expensive. Since it is a fertile nuclear material, it's use and distribution is under the control of the federal government. Titanium oxide is readily reduced to lower oxides and cannot be used in neutral or reducing atmospheres.

Carbon, Carbides, and Nitrides. Carbon (graphite) is a good thermal and electrical conductor. It is not easily wetted by chemical action, which is an important consideration for corrosion resistance. It tends to oxidize at high temperatures, but can be used up to 2760 ^{0} C for short periods in neutral or reducing conditions. Upon exposure to higher temperatures, graphite has the potential to produce volatile hydrocarbons which prevents its use in this application. When heated under oxidizing conditions, silicon carbide and silicon nitride, Si_3N_4, can be used up to 1100 ^{0} C. In reducing or neutral atmospheres, their useful range is much higher. Silicon carbide has very high thermal conductivity and can withstand thermal shock cycling without damage. It is also an electrical conductor and is used for electrical heating elements. Other carbides have relatively poor oxidation resistance, but under neutral or reducing conditions, they have potential usefulness as technical ceramics in aerospace application. For example the carbides of B, Nb, Hf, Ta, Zr, Ti, V, Mo, and Cr. Ba, Be, Ca, and Sr are hydrolyzed by water vapor.

Silicon nitride has good strength retention at high temperature and is the most oxidation resistant nitride. Boron nitride has excellent thermal-shock resistance and is in many ways similar to graphite, except that it is not an electrical conductor.

Silicon Carbide. Silicon carbide is made by the electrofusion of silica sand and carbon. Silicon carbide is hard, abrasion resistant, and has a high thermal conductivity. It is relatively stable but has a tendency to oxidize above 1400 ° C. The silica thus formed affords some protection against further oxidation.

Beryllia and Thoria. These are specialty oxides for highly specialized applications that require electrical resistance and high thermal conductivity. Beryllia is highly toxic and must be used with care. Both are very expensive and are used only in small quantities.

Metals. The highest-melting refractory metals are tungsten (3400 ° C), tantalum (2995 ° C), and molybdenum (2620 ° C); all show poor resistance to oxidation at high temperatures. Hafnium-tantalum alloys form a tightly adhering oxide layer that gives partial protection up to 2200 ° C; the layer continues to grow with extended use.

Alumina ceramics are the most widely used oxide-type ceramic, chiefly because alumina is plentiful, relatively low in cost, and equal to or better than most oxides in mechanical and thermal conductive properties. Density can be varied over a wide range, as can purity-down to about 90% alumina to meet specific application requirements. Alumina ceramics are the hardest, strongest, and stiffest of the oxides. They are also outstanding in electrical resistivity and dielectric strength, are resistant to a wide variety of chemicals, and are unaffected by air, water vapor, and sulfurous atmospheres. With a melting point of only 3700 ° F (2037 ° C), they are relatively low in refractoriness, and at 2500 ° F (1371 ° C), retain only about 10% of room-temperature strength. The chief uses for alumina are for the production of aluminum metal and for abrasives, but it is also used for ceramics, refractories, pigments, catalyst carriers, and in chemicals. Aluminum oxide crystals are normally hexagonal, and are minute in size. The specific gravity is about 3.95, and the hardness is up to 9 Mohs. The alumina powder is of two kinds. Type A is *alpha alumina* with hexagonal crystals with density 4.0, and hardness 9 Mohs. Type B is *gamma alumina* with cubic crystals, smaller particle size, specific gravity of 3.6, and hardness 8. At high temperatures gamma alumina transforms to the alpha crystal. The aluminum oxide most frequently used for refractories is the *beta alumina* in hexagonal crystals heat-stabilized with sodium. *Activated alumina* is partly dehydrated alumina trihydrate, which has a strong affinity for moisture or gases and is used for dehydrating organic solvents. *Activated*

alumina F-1, of the Aluminum Co. of America, is a porous form of alumina, used for drying gases or liquids. It will remove moisture up to 15% of the dry weight of the alumina. This affinity for holding moisture allows it to be used as a two-faceted coolant for the aerosol. The cooling characteristics of water retained in the porous alumina media allow for added thermal dissipation at lower temperatures where the alumina solid is less effective. These porous media in a packed-bed configuration provide good heat exchange and thermal absorption.

TABLE 2. THERMODYNAMIC PROPERTIES OF REFRACTORY MATERIALS

	Specific Heat (KJ/Kg K) @ 600 K	Melting Point (C)	Enthalpy of Fusion (KJ/mole)
Aluminum Oxide	1.09	2054	78
Magnesium Oxide	1.17	2826	111

G. THERMAL TRANSFER IN AEROSOL SUSPENSIONS

The study of thermal transfer in aerosol suspension flow has been in areas such as various types of catalytic reactors in chemical engineering and solid propellant rocket fuel. This research can be utilized in the determination of convective and radiative heat transfer of the EMAA aerosol to refractory media in packed bed configuration. Radiative heat transfer becomes the dominant heat transfer mode especially within a high temperature region. The suspended solid particles emit and absorb the thermal radiation directly from EMAA combustion and exchange it with the surrounding gas through conduction and convection. The turbulent structure of the heated gas stream is substantially changed through the addition of the micron-size EMAA particles. This effect has both positive and negative sides depending on the situation. If sufficiently coarse particles are suspended, then the wake may be formed behind each particle and this in turn leads to the enhancement of the turbulent level of the flow and therefore of the convective heat transfer. In the case of the EMAA combustion and aerosol production, finer particles tend to calm the fluctuation of the flow through viscous drag on their surfaces and also through their dividing the turbulent eddies into smaller ones, which leads to a higher dissipation rate of the turbulent energy, and a corresponding reduction in the convective heat transfer. (Due to the small

size of the particles, heat transfer resistance between the particles and loading gas is usually minor and the two substances can be assumed to be in thermal equilibrium.)

H. PACKED-BED COOLING

The use of heat absorbing materials to remove heat from the aerosol stream follows from the need of removing heat at a similar rate of that which was generated. Solids have, in general, better thermal conductivity than water, but not as good thermal heat storage capacity. Another important property is that of having a high melting point so as to maintain structural integrity of the device. The principle of cooling is based on allowing the aerosol stream to flow through a bed of these heat-absorbing materials. The packing of the bed is critical as the optimum surface area of the refractory media is key to an effective heat transfer from the stream to the heat-absorbing material. The surface area of the refractory media allow for a convective surface area, and thus the heat-transfer rate is largely dependent on their packing and available surface area. The packing of large area media, besides adding weight to the device, allows for a less constricted flow thus increasing the total path required for a given amount of energy removal; less energy transfer per unit path length. Conversely, a packed-bed made up of smaller media would present an even larger surface area exposure to the aerosol per unit path length. Thus smaller surface area media would remove more energy per unit path length from the flow.

However, an additional concern is the adhesion of aerosol particles to the surface of the media. Larger spherical media packed in a bed offer a smaller area to convective heat transfer to the aerosol stream and thus less adhesive area for the aerosol. In the case of smaller media, the effective convective area is larger and thus the effective area for adhesion of the aerosol is also large. Thus an optimization process whereby the surface area and overall total volume of the media required to remove heat from the aerosol stream is required.

I. AEROSOL FLOW CHARACTERISTICS IN PACKED-BED CONFIGURATIONS

The design procedure of the EMAA aerosol generator begins with the estimation of pressure drop along the flow path. The pressure drop data is of interest because it gives insight into the turbulence mechanism of the aerosol suspension flows. Methods of correlating the pressure drop data of the aerosol suspension flows differ because several different procedures are utilized. When comparing experimental results, attention should be given to which procedure is used. The first method correlates pressure drop data according to the following equation,

$$\Delta P_m / \Delta P_o = 1 + \Gamma \cdot m \tag{4}$$

where Γ represents the ratio of the mass flow rate of the particulate phase to that of the gaseous phase, usually referred to as the solid loading ratio, while ΔP_m and ΔP_o are the pressure drops of the following line of specified length L with and without the suspended phase, respectively. In this procedure, constant m must be determined experimentally and may be a function of geometric properties of the suspended particles, pipe roughness, gas Reynolds number, and others. The universality of this correlation is restricted though, because a significant part of the measured pressure drop is due to the gravity force exerted on the suspended particles. The flow conditions would be quite varied between vertically upward and downward flows and both of them would also be different from the horizontal flows.

In other procedures which do not use equation (4), the measured (total) pressure drop is divided into several contributions. It is expressed as,

$$\Delta P_m = \Delta P_{af} + \Delta P_{ap} + \Delta P_{pf} + \Delta P_{ff} + \Delta P_{fp} \tag{5}$$

The first two terms of the right-hand side represent the pressure drops due to acceleration of the gaseous phase and the solid phase, respectively. Therefore both can be neglected when the flow is fully accelerated or developed. The next two terms of the equation are the pressure drops necessary to sustain the columns of both phases or simply static heads of both phases. The signs before these terms should be changed in case of vertically downward flows. The last two terms of the equation represent frictional pressure drops of both phases. In the case of the EMAA combustion the method for correlating pressure drop data is based on this equation and derives

the following equation for the frictional pressure drop of the particulate phase in connection with the pressure drop of packed beds,

$$\Delta P_{ff}/L = \alpha^2/(1-\alpha)^3 \, \mu \cdot U_f/d_p^2 + 1.75 \, \alpha/(1-\alpha)^3 \, \rho \, D_f U_f^2/d_p \qquad (6)$$

where U_f, μ, d_p and ρ_f denote gas velocity, gas viscosity, particle diameter, and gas density, respectively. The volume fraction of the suspended phase α (=1 - ε ; ε = voidage) appears in this equation. For the packed beds, the estimation of α or ε is simplified and it is usually fixed at a certain value given the use of spherical media in the packed-bed configuration.

J. PACKED-BED REFRACTORY THERMODYNAMICS

The design and fabrication of the EMAA aerosol generator utilized background research of refractories for thermal exchange and performance data of EMAA combustion. For EMAA formulation A the maximum achieved temperature during combustion is approximately 2500 ^{0} F (1371 ^{0} C). Ideally the goal for the generator is to cool the aerosol to approximately 160 ^{0} F (71 ^{0} C) at discharge. From previous tests, the heat generated upon combustion for EMAA formulation A is 955 cal/gram, which provides 477,500 calories from a 500 gram charge designed to provide coverage for a 10 cubic meter volume. If water alone were used as a coolant, the amount necessary would be the amount of heat (477,500 cal) divided by the latent heat of water (542 cal/gram @ 1 atm @ 100 ^{0} C) or 881 grams of water (1.9 lb). Unfortunately, because water vaporizes so quickly at these temperatures, it alone is not a suitable candidate.

As previously discussed, alumina (Al_2O_3) is an excellent refractory. The measure of alumina's ability to cool the aerosol is measured by its specific heat at constant pressure, c_p. This is defined as the quasi-static heat flux per mole required to produce unit increase in the temperature of a system maintained at constant pressure. Using the c_p of alumina at STP (0.26 cal/gm K) and the overall system temperature change (1371 - 71) K or 1300 K the heat transferred to the refractory is 338 cal/gm. If the total system heat is defined by Q, then the amount of alumina needed as refractory material is Q (477,500 cal) divided by the specific heat c_p (0.26 cal/gm K) times the

change in temperature (1300 K). Thus the mass of pure alumina required is 1413 gm (3.1 lb). Utilizing the activated, high-porosity alumina media allows for moisture to be contained within, and provides better thermal absorption at both low and high temperatures. The porous media are 42% by weight water, so the mass of refractory to absorb Q (477,500 cal) is different. 1.42 gm of activated alumina contains 1.0 gm Al_2O_3 and 0.42 gm water. This is 338 cal/gm for 1.0 gm alumina and 228 cal/gm for 0.42 grams of water. This equals 400 cal/gm of activated material, so the total weight needed is Q divided by 400 cal/gm, or 1194 gm (2.6 lb). Given the properties of the solid alumina and activated alumina, the packed-bed configuration uses both types, with 100% solid near the combustion chamber, 50% water activated in the middle of the packed bed, and 100% water activated where the aerosol has already been significantly cooled and near the exit.

K. GENERATOR HOUSING

Primary considerations for the generator housing are heat resistance, and containment of the flame and potential pressure buildup produced from EMAA combustion. Steel provides the benefits of heat resistance, availability, strength, and recyclability or potential reuse. Steel tubing is also readily available in various-size diameters and thicknesses, is easily machined, and provides adequate long-term corrosion resistance.

The design for the 500 gram EMAA charge generator utilizes a 13 inch stainless steel tube with a 5 inch outside diameter, and 0.12 inches thickness. The sealed end of the housing uses 4 bolts, evenly spaced, and welded to the interior wall of the tube so as to secure a 0.125 inch thick plate. This plate has a machined lip to provide a uniform seal when bolted, and also has a central port with the necessary diameter to connect the ignitor squib at the EMAA charge to the ignition system. Beneath the plate is a 1.5 inch gap, which provides a buffer-zone between the top of the EMAA charge housing and the interior of the plate. This housing consists of 2.25 inch section of 3.0 inch outside diameter stainless steel tubing centered within the generator. The housing rests on 0.1 inch stainless steel mesh secured by two 0.125 inch stainless rods connected to the generator body. The space between the charge housing, the top plate, and the generator body is

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filled with high-temperature resistant alumina fiber blanket, which is easily contoured to fill these voids. This blanketing acts to reduce flame contact with the generator wall, and provides insulation and added thermal transfer.

Upon combustion, the aerosol flows through 9.0 inches of the packed-bed configuration of 0.25 inch alumina spheres, cooling it before it exits via an identical stainless steel mesh arrangement at the open end of the generator. Even with the large amount of void space in the packed-bed and relatively unrestricted flow from the charge housing to the outside space, possibility of pressure buildup from combustion should be taken into consideration.

L. IGNITION SYSTEM

Ignition of the EMAA charge within the combustion chamber utilizes a temperature specific thermocouple. This thermocouple uses two dissimilar metals that produce a current when heated. This current completes a circuit between a 9V battery and a magnesium ignitor squib which is inserted into the primer of the EMAA charge. In addition, a fail-safe ignitor should be considered, possibly a paraffin-coated woven cord running into the EMAA primer. In the case of a failed electrical ignition, this would provide fuse-type ignition of the EMAA charge.

M. TESTING AND RESULTS

Testing of the various generator devices was conducted at the Total Flood Test Facility, Tyndall AFB, Florida. Testing was designed to test both fire extinguishing capabilities and safety of utilization. Principle safety concerns include dissipating generated heat to reduce possibility of secondary fire, and reducing pressure accumulation to prevent breaching the generator housing. Comparing the temperature histories of the generator designs shows progress to an acceptable level cooling. Testing of the initial generator, developed to show the effects of a delivery system with virtually no heat absorption and very little hindrance of generated aerosol, shows a peak temperature of 843 ^{0} C at 90 seconds burn time (Figure 10).

The water-cooled design developed a peak temperature of 566 ^{0} C at 55 seconds burn time (Figure 11). The use of refractories lowered the peak temperature to 90 ^{0} C at 120 seconds burn time with a combined alumina and magnesia mixture in a packed-bed configuration (Figure 12). The use of only magnesia resulted in a peak temperature of 60 ^{0} C at 160 seconds burn time (Figure 13), but the coarseness and irregular shapes of the magnesia refractory restricted aerosol flow and presented potential problems with aerosol deposition and pressure increase. Subsequent testing with larger capacity generators, one and two kilogram SFE charge capacity, showed efficient heat absorption, (Figures 14 and 15), with the peak temperature of the one kilogram charge generator 66 ^{0} C at 175 seconds burn time, and the two kilogram charge generator 93 ^{0} C at 87 seconds burn time.
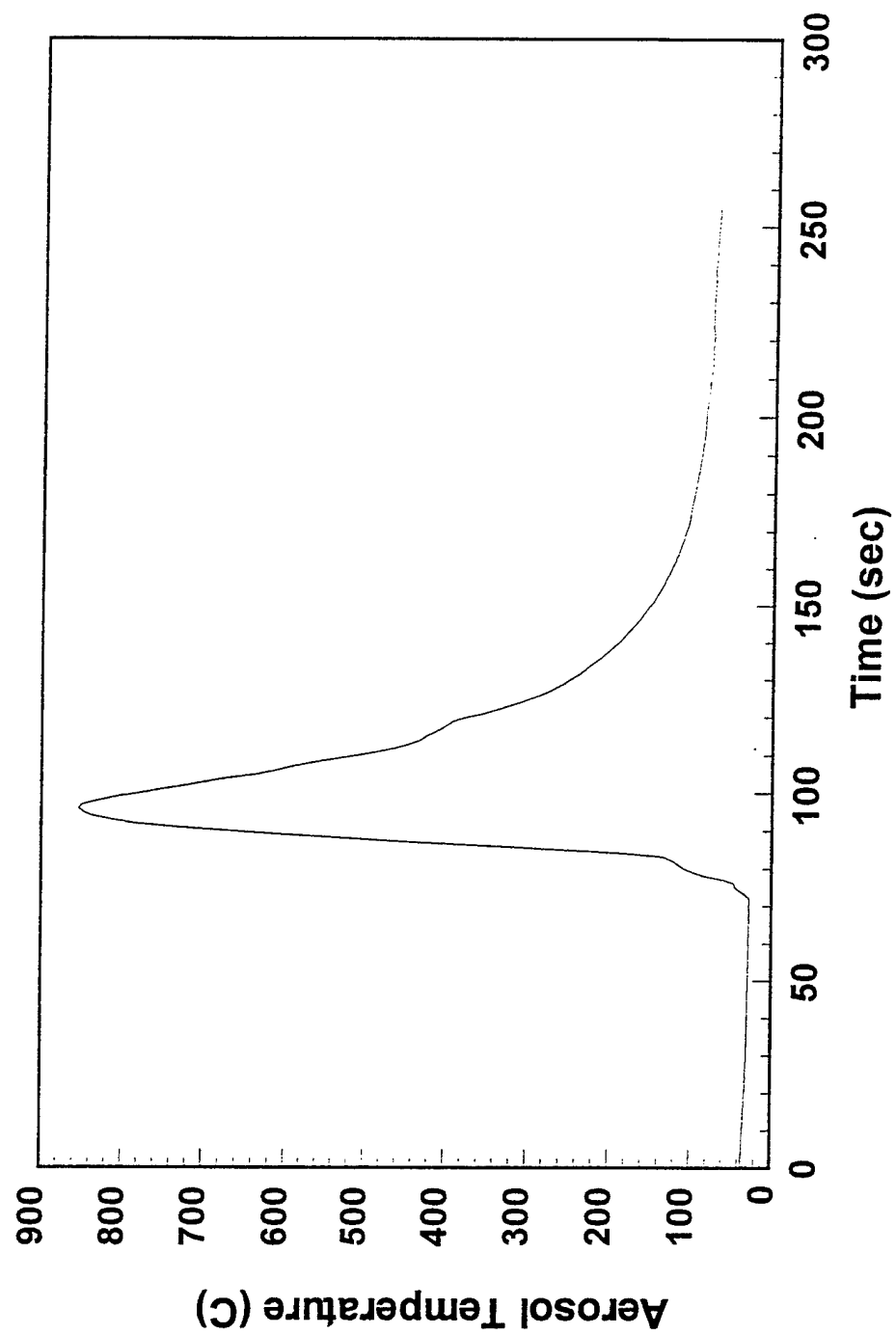
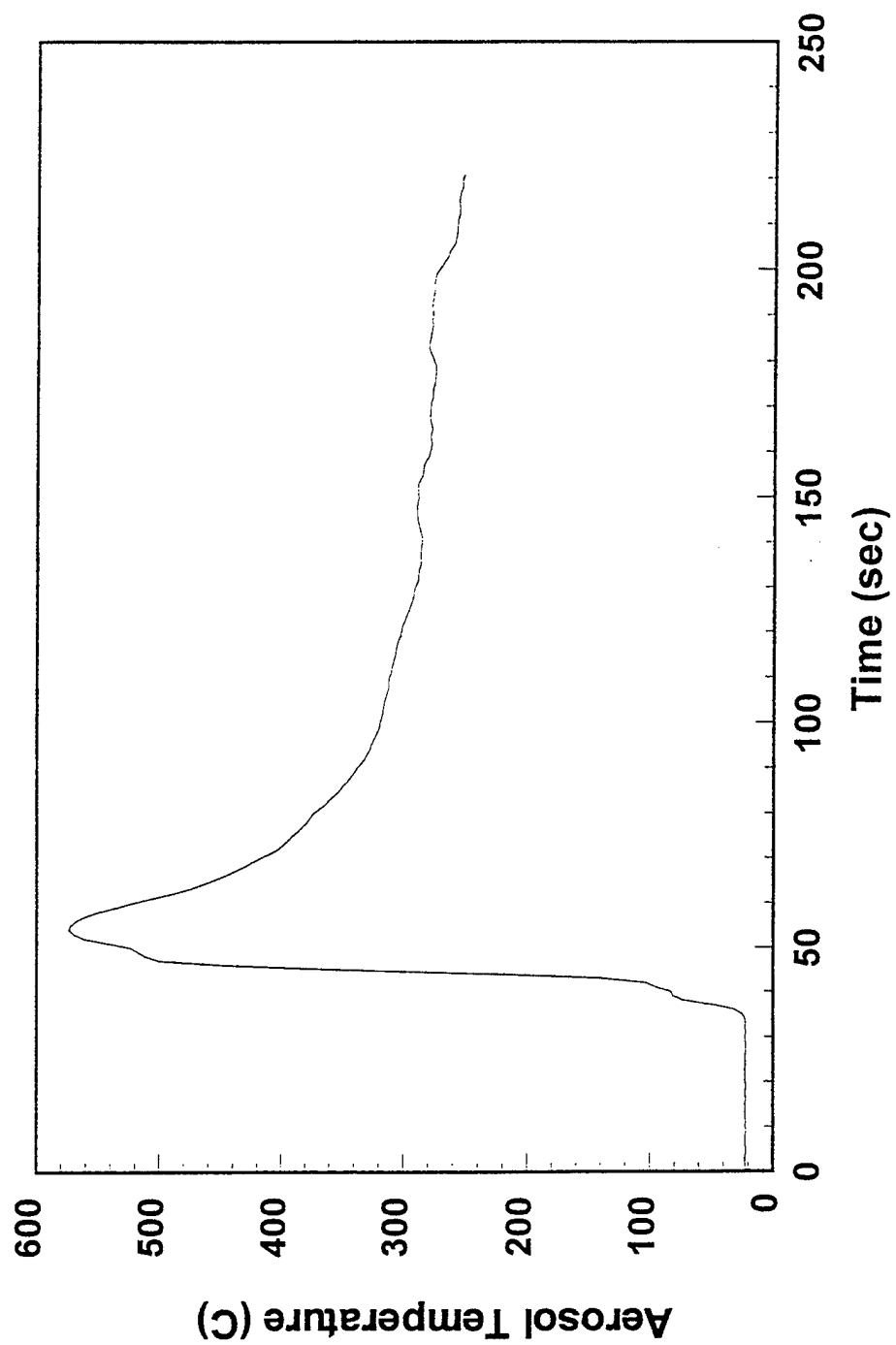Figure 10. Temperature History of Aerosol Discharge into Open Air.

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Figure 11. Temperature History of Aerosol Generator Discharge into Delivery System Composed of a Water Jacket.

Figure 12. Temperature History of Aerosol Generator Discharge into Delivery System Composed of a Packed Bed of Refractory Materials (Magnesia and Alumina).

Figure 13. Temperature History of Aerosol Generator Discharge into Delivery System Composed of a Packed Bed of Magnesia.

Figure 14. Temperature History of Aerosol Generator Discharge into Delivery System Composed of a Packed Bed of Refractory Materials, 1 Kg SFE.

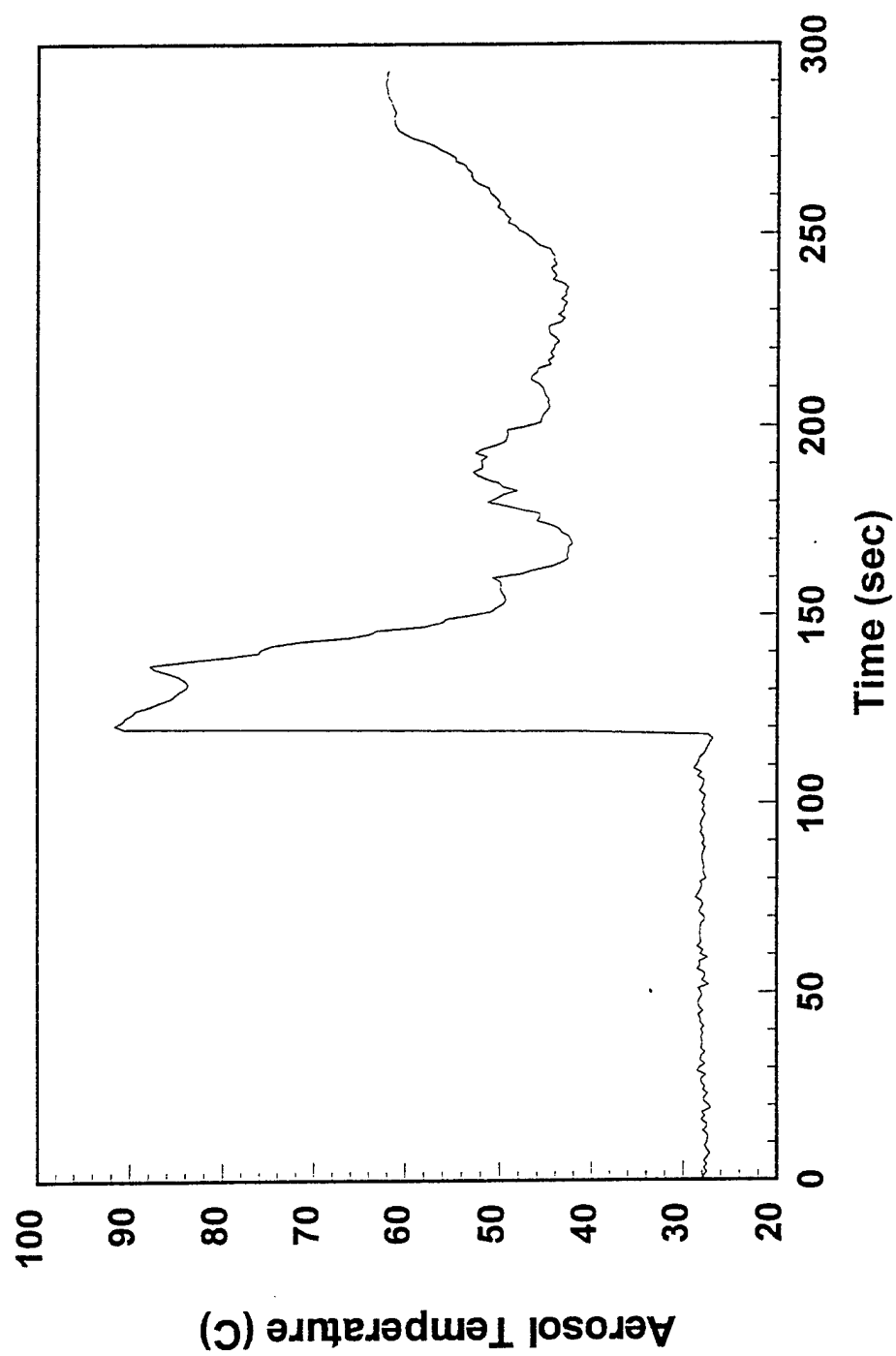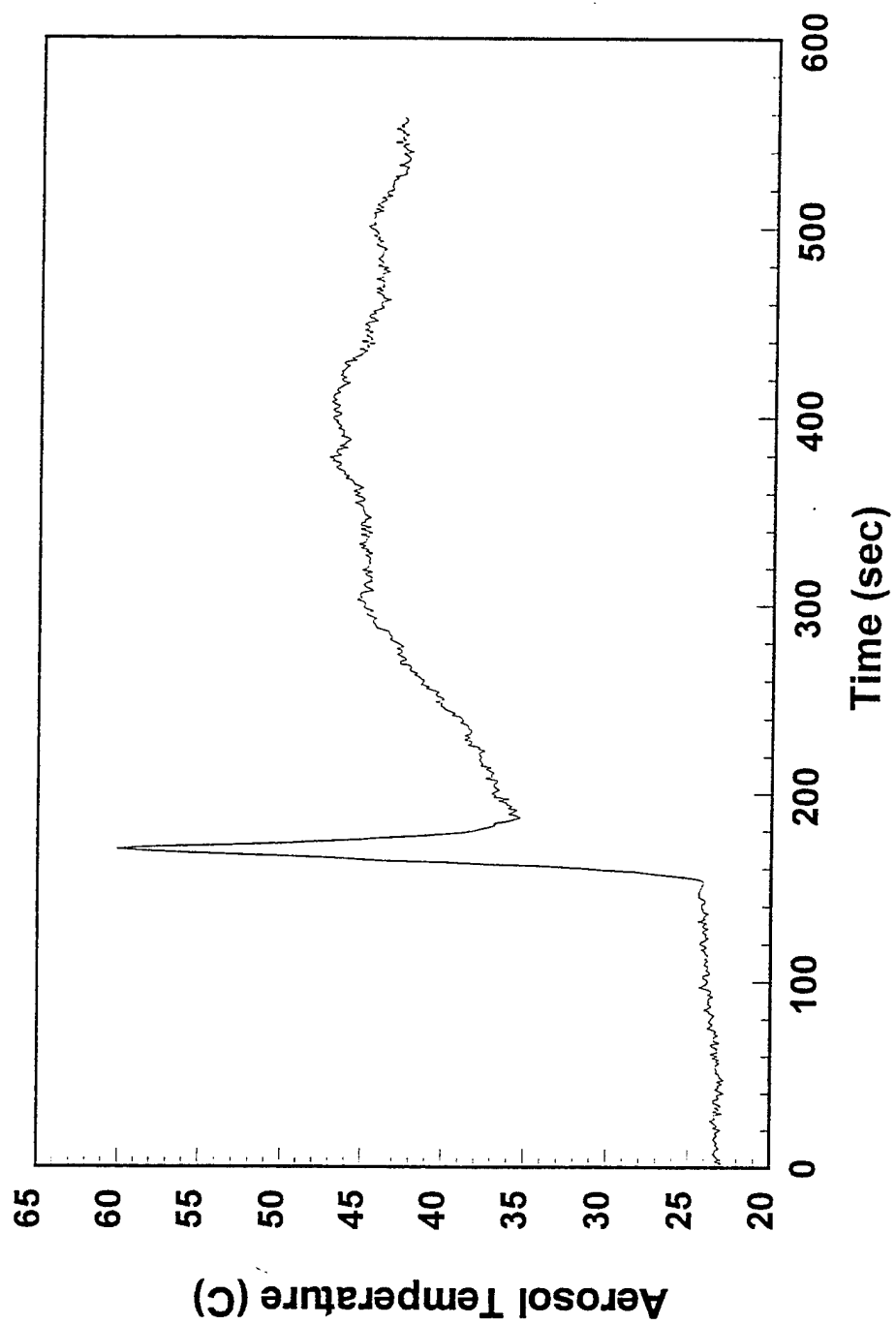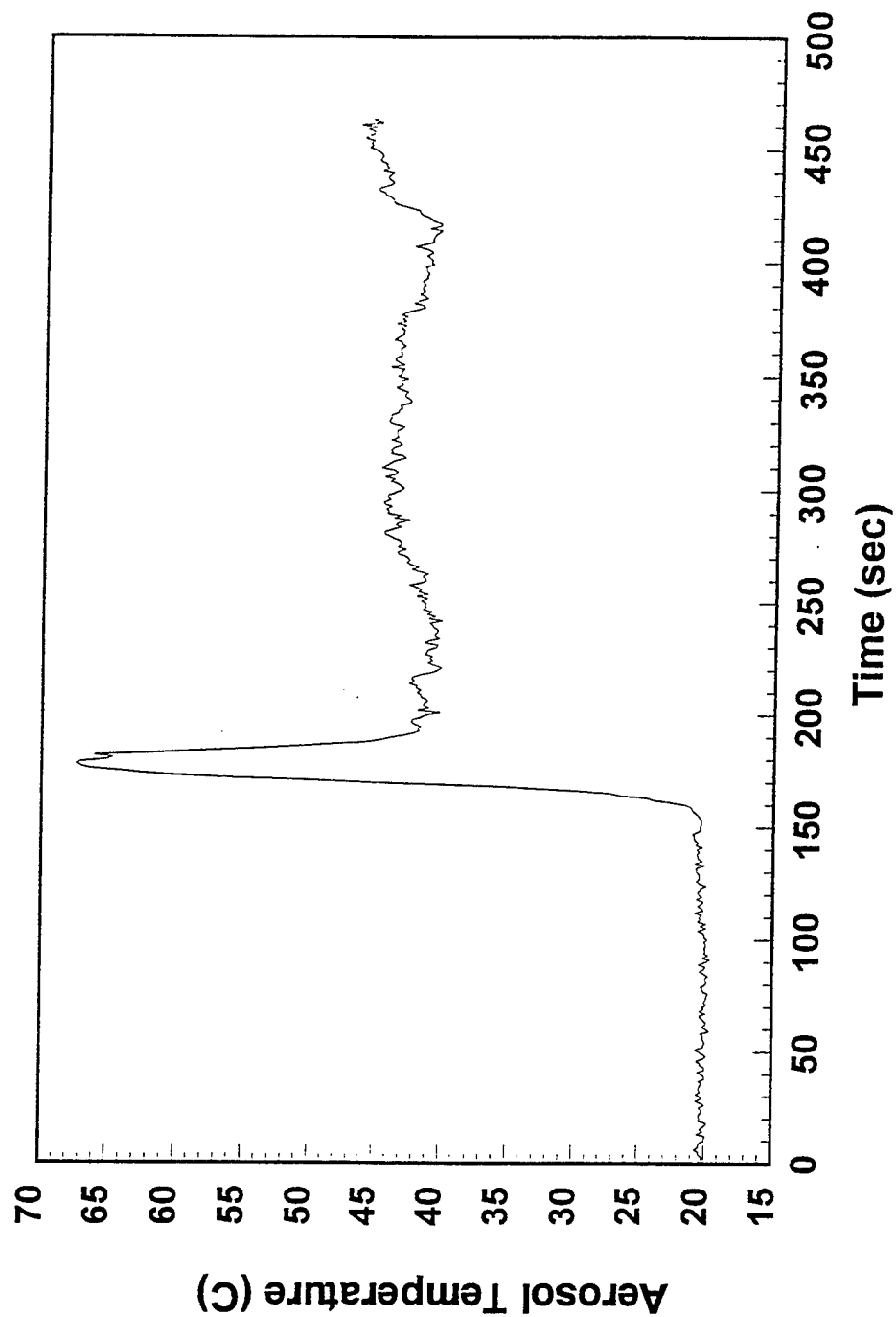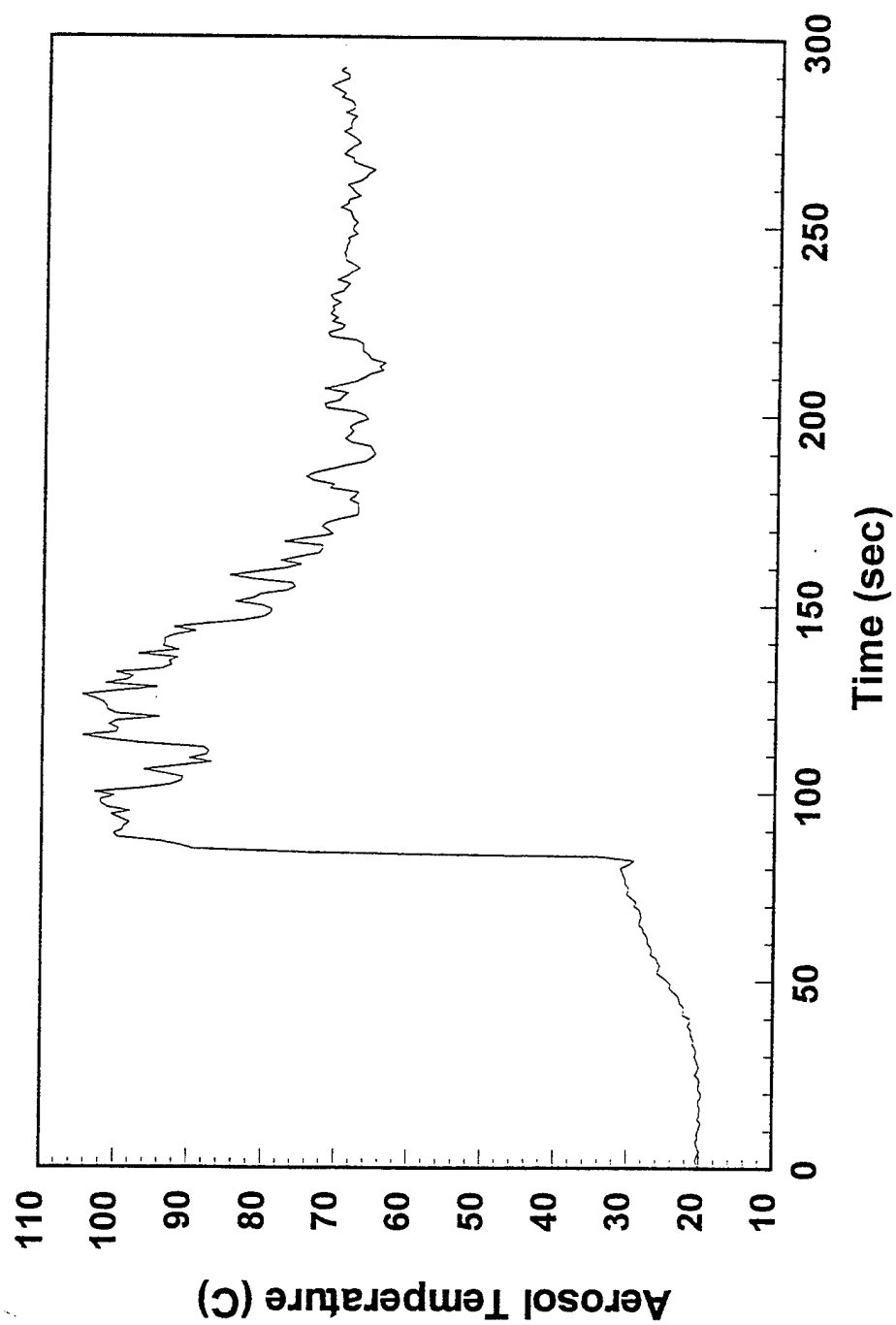**Figure 15. Temperature History of Aerosol Generator Discharge into Delivery System Composed of a Packed Bed of Refractory Materials, 2 Kg SFE.**

SECTION IV

CORROSION STUDY

A.BACKGROUND

Corrosion of metals and metal alloys has been studied in both simulated and actual environments. Because of the many factors that influence corrosion in metals (e.g., humidity, temperature, presence of corrosive agents, airborne pollutants, wind direction, material surface finish), a simulated environment is preferred when trying to identify the corrosive actions of a single variable. Results of past studies have applications principally in industry, where corrosive agents may be utilized in a manufacturing process. There have also been considerable studies on exposed metals in various environments, marine, industrial, and others. These must be taken into account when considering what is already occurring before introduction of a corrosive. Corrosion studies take into account different factors of corrosion which potentially damage metal surfaces (e.g., overall mass loss, pitting frequency, depth and shape of pitting, time to perforation). It is important to note that studies provide data that shows tendencies of corrosives that may be exacerbated by one or more environmental conditions.

B. INTRODUCTION

One of the concerns regarding the use of the pyrotechnically generated aerosol is how corrosive they may be due to prolonged contact with various metals and metal alloys. Depending upon the composition of the starting materials, the pyrotechnically produced aerosol can consist of potassium chloride (KCl), potassium hydroxide (KOH), and/ or magnesium hydroxide (MgOH). Considering the many ways the aerosol may be utilized, it is possible for the potentially corrosive alkali salts produced upon combustion to come in contact with a variety of different metal surfaces. For this reason, this preliminary study provides an assessment of

corrosion damage on samples of ten commonly used metals and metal alloys. This report provides data and results from a study lasting approximately 30 days. It should not be extrapolated to predict results for longer times of exposure, because corrosion does not always occur uniformly with respect to time. Some metals form passive films that indicate high initial corrosion rates, but after film formation, these rates fall dramatically. Prolonged testing of these types of metals and metal alloys allows for these films to be broken down, and more accurate corrosion assessment to occur. A suggested guideline for test duration from the American Society of Testing and Materials (ASTM) is: duration (hours) = 2000/(anticipated corrosion rate in mils per year). By using a test period of approximately 30 days for EMAA, a reasonably good assessment of corrosion damage may be predicted. It should also be noted that indoor environments, atmospheric conditions, airborne pollution, temperature, wind direction, and design features, all potentially affect corrosion types and rates. For this reason results should be considered indicative of potential corrosion rates and types.

C. TYPES OF CORROSION

Corrosion is an electrochemical process that results in the degradation of a metal or alloy. It is a coupled reaction between an anode and at least one cathode. Corrosion (oxidation) occurs at the anode while reduction occurs at the cathode. The various ways (forms) that corrosion manifests itself are myriad but can often be reduced to several basic types.

Uniform/General Corrosion: A form of attack that produced overall uniform wastage of the metal. Often associated with atmospheric corrosion and some high temperature oxidation or sulfidation attack.

Pitting Corrosion: A high localized attack of the metal creating pits of varying depth, width, and number. Pitting may often lead to complete perforation of the metal with little or no general corrosion of the surface.

Crevice Corrosion: Similar to pitting corrosion in its localized nature but associated with crevices. Stainless steels and some nickel-base. alloys are particularly susceptible to this form of corrosion.

Dealloying: The selective removal of one element (usually the least noble) from an alloy by the corrosive environment. Also referred to as selective leaching or dezincification, denickelification, etc. designating the element removed.

Intergranular Attack: The preferential corrosion of grain boundaries in a metal caused by prior thermal treatments and related to specific alloy chemistries.

Corrosion Fatigue: The initiation and extension of cracks by the combined action of an alternating stress and a corrosive environment. The introduction of a corrosive environment often eliminates the fatigue limit of a ferrous alloy creating a finite life regardless of stress level.

Erosion-Corrosion: Many forms of flow assisted corrosion are often included in this term such as cavitation, impingement, and corrosion-erosion. All of these types of attack are the result of accelerated corrosion due to flow of solids, liquids or gases.

Galvanic Corrosion: Accelerated corrosion of the least noble metal when coupled to one or more other metals. The more noble metals are protected from corrosion by this action.

Stress Corrosion Cracking: The initiation and propagation of cracks by the combined action of a corrosive environment and a tensile stress. Generally, susceptibility to cracking increases with increasing temperature. Not every alloy cracks in every environment, however, the list of environment/ alloy combinations that produce stress corrosion cracking is continually increasing.

Hydrogen Damage: There are numerous forms of damage associated with hydrogen which are contained under the collective term "hydrogen damage." For hydrogen embrittlement and hydrogen stress cracking, a tensile stress and hydrogen atoms are necessary to cause failure. However, contrary to stress corrosion cracking, susceptibility is greatest near room temperature. Other terms and forms are: hydrogen induced cracking, blistering, sulfide stress cracking, hydrogen stress corrosion cracking, hydriding, hydrogen attack. There are many others too numerous to mention.

Although these forms are presented in the context of aqueous corrosion, many of them are also operative at high temperature. High temperature corrosion by oxidation and sulfidation can take the form of uniform attack, pitting, or dealloying to name a few. It is of utmost importance to recognize the difference between high temperature corrosion (oxidation) and aqueous corrosion.

The mechanisms are different and therefore, the correct alloy choice for resistance to one environment often is incorrect if applied to another. Most corrosion rates are reported as a time averaged weight loss (mils per year, millimeters per year) that implies a uniform corrosion rate. However, in actual fact, few metals and alloys corrode in a uniform manner rather, highly localized corrosion (pitting, crevice corrosion, etc.) is the rule. In simple terms, corrosion can be controlled by any of four methods: cathodic protection, coatings., inhibitors and/or alloy changes, often used in combination with alloys of lower corrosion resistance to retard or eliminate corrosion.

D. ATMOSPHERIC FACTORS AFFECTING CORROSION

Atmospheres are often classified as being rural, industrial, or marine in nature, but this is an oversimplification. For example, there are locations along the coast that have heavy industrial pollution in the atmosphere, and they exhibit characteristics that are both marine and industrial. Expansion into formerly rural areas can easily change the aggressiveness of a particular location. Finally, long-term trends in the environment, such as changes in rainfall patterns, mean temperature, and perhaps acid rain, can make statistics from past behavior less reliable. Other factors that limit the usefulness of atmospheric exposure data are the general non-uniformity of weight loss due to corrosion with time and the fact that most atmospheric corrosion data are presented as an average over the entire test panel surface. Most atmospheric exposure data for steels show a decrease in the rate of attack with time of exposure so that extrapolations of such data to times longer than those covered by the exposure data can lead to erroneous corrosion rate predictions. Finally, in many cases, the average weight loss per unit area is of less concern than the time to perforation. This factor is more related to localized attack, which can be masked by the averaging of data, as is done in weight loss determinations.

Given these variables, using atmospheric-corrosion data should be more indicative than quantitative. Perhaps most important it should be remembered that it is impossible to describe either the extent or rate of corrosion under atmospheric conditions with a single parameter, which is what much of the reported corrosion data persists in doing. When the results of a several-year

exposure test are condensed to a single value, such as the average loss per year or the total loss for the exposure period, one cannot estimate the values of the kinetic parameters governing the system. Without the values of these parameters, extrapolation of results to longer exposure periods becomes quite unreliable. When good estimates for the kinetic parameters are available, extrapolations to 7- or 8-year performance from 1- to 2-year data have been found to agree within 5% of observed performance.

E. MATERIAL SUMMARIES

The following summaries were compiled from a survey of the available literature. This information is included to act as a basis for determining the level of corrosion in various environments that metals may undergo before exposure to EMAA formulations.

1. Aluminum

Most aluminum alloys have been shown to be resistant to atmospheric corrosion in laboratory tests. Alclad aluminum roofing and siding have been employed in a wide variety of industrial atmospheres. In such applications, corrosion is arrested at the cladding/core interface due to cathodic protection by the 1% Zn aluminum alloy cladding. Large quantities of these Alclad aluminum sheet products have been used in port facilities throughout the United States in applications such as transit sheds, storage buildings, and the like. Aluminum has been used for electrical power cables since early in this century. Corrosion of most aluminum alloys by weathering is restricted to mild surface roughening by shallow pitting. However, such attack is more severe for alloys with higher copper contents, and such alloys are seldom used in outdoor applications without protection. Corrosivity of the atmosphere to metals varies greatly from one geographic location to another, depending on such weather factors as wind direction, precipitation and temperature changes, amount and type of urban and industrial pollutants, and proximity to natural bodies of water. Service life may also be affected by the design of the structure if weather conditions cause repeated moisture condensation in crevices or in channels with no provision for drainage. Laboratory exposure tests, such as salt spray, total-immersion tests, provide useful comparative information, but have limited value for predicting actual service

performance and sometimes exaggerate differences among alloys that are negligible under atmospheric conditions

Effect of Exposure Time. A very important characteristic of weathering of aluminum and of corrosion of aluminum under many other environmental conditions is that corrosion rate decreases with time to a relatively low, steady-state rate. This deceleration of corrosion occurs regardless of alloy composition, type of environment or the parameter by which the corrosion is measured. However, loss in tensile strength, which is influenced somewhat by pit acuity and distribution, but is basically a result of loss of effective cross section. The decrease in rate of penetration of corrosion is dramatic. In general, rates of attack at discrete locations, which is initially about 0. 1 mm/yr (4 mpy), decrease to much lower and nearly constant rates within a period of about 6 months to 2 years. For the deepest pits, the maximum rate after about 2 years does not exceed about 0.003 mm/yr (0.1 mpy) for severe seacoast locations and may be as low as 0.0008 mm/yr (0.03 mpy) in rural or and climates.

2.   Carbon Steels

The effects of various atmospheres on the corrosion rates of cold-rolled carbon steels were detailed in a series of weight-loss measurements performed after 2 years of exposure. The most revealing feature of this study was the extreme range of corrosion rates occurring at the various test sites. This difference in corrosion rate is easily greater than any effect that can be produced by small changes in composition of the steel. Again, this underscores the fact that with the corrosion of carbon steels the alteration of design or environmental factors is usually more effective than changing the grade of steel. Because carbon steels are by definition not very highly alloyed, most grades do not exhibit large differences in atmospheric corrosion rates. Nevertheless, alloying can make changes in the atmospheric corrosion rate of carbon steel. The elements generally found to be most beneficial in this regard are copper, nickel, silicon, chromium, and phosphorous. Of these, the most example is that of copper, increases from 0.01 to 0.05% have been shown to decrease the corrosion rate by a factor of two to four. Additions of the above elements in combination are generally more effective than when added singly, although the effects are not additive. The effectiveness of these elements in retarding corrosion

also appears to depend on the corrosive environment with the most benefit it appearing in industrial atmospheres.

3.   Alloy Steels

A study of 270 alloy steels was performed in which experimental heats of steel involving systematic combinations of chromium, copper, nickel, silicon, and phosphorus were tested to determine the individual and joint contributions of these elements to corrosion resistance. The data showed that long-term atmospheric corrosion of carbon steels can be reduced by small additions of copper, that additions of nickel are also effective, and that chromium in sufficient amounts is helpful if copper is present. The maximum resistance to corrosion was obtained in this study when alloy contents were raised to their highest levels. Some of the results obtained in industrial environments were as follows. The corrosion rate for carbon steel becomes constant after about 5 years. The corrosion rate for copper steel levels off to a constant value after about 3 years. High-strength low-alloy steel, which contains several alloying elements, exhibits a constant rate after approximately 2 years, and corrosion of this steel eventually ceases. ASTM low-alloy steels exhibit significantly better performance than either carbon steel or structural copper steel. Although the results given above provide good estimates of average corrosion behavior, corrosion rates can increase significantly in severe environments. This study does, however, demonstrate the effectiveness of increased alloy content on corrosion resistance.

4.   Stainless Steels

The atmospheric contaminants most often responsible for the rusting of structural stainless steels are chlorides and metallic iron dust. Chloride contamination may originate from the calcium chloride ($CaCl_2$) used to make concrete or from exposure in marine or industrial locations. Iron contamination may occur during fabrication or erection of the structure. Although marine environments can be severe, stainless steels often provide good resistance. In a study comparing several AISI 300-series stainless steels after a 15-year exposure to a marine atmosphere 250 m (800 ft) from the ocean at Kure Beach, North Carolina, materials containing molybdenum exhibited only extremely slight rust stain, and all grades were easily cleaned to reveal a bright surface. Type 304 stainless steel may provide satisfactory resistance in many marine

applications, but more highly alloyed grades are often selected when the stainless is sheltered from washing by the weather and is not cleaned regularly. Type 302 and 304 stainless steels have had many successful architectural applications. Type 430 stainless steel has been used in many locations, but there have been problems. For example, type 430 stainless steel rusted in sheltered areas after only a few months of exposure in an industrial environment The type 430 stainless steel was replaced by type 302, which provided satisfactory service. In more aggressive environments, such as marine or severely contaminated atmospheres, type 316 stainless steel is especially useful.

5.   Coppers

Copper and copper alloys are suitable for atmospheric exposure. Copper and copper alloys resist corrosion by industrial, marine, and rural atmospheres, except atmospheres containing NH_3 or certain other agents where stress-corrosion cracking has been observed in high-zinc alloys (>20% Zn). The severity of the corrosion attack in marine atmospheres is somewhat less than that in industrial atmospheres, but greater than that in rural atmospheres. However, these rates decrease with time. Individual differences in corrosion rates do exist between alloys, but these differences are frequently less than the differences caused by environmental factors. Thus, it becomes possible to classify the corrosion behavior of copper alloys in a marine atmosphere into two general categories: those alloys that corrode at a moderate rate and include high copper alloys, silicon bronze, and tin bronze; and those alloys that corrode at a lower rate and include brass, aluminum bronze, nickel silver, and copper nickel. Environmental factors can cause the median thickness loss to vary by as much as 50% or more in a few extreme cases. Those environmental factors that tend to accelerate metal loss include high humidity, high temperatures (either ambient or due to solar radiation), proximity to the ocean, long times of wetness, and the presence of pollutants in the atmosphere. The converse of these conditions would tend to retard metal loss. Metallurgical factors can also affect metal loss. Surface finish also plays a role in that a highly polished metal will corrode slower than one with a rougher surface. Finally design details can affect corrosion behavior. For example, designs that allow the collections of rainwater will often exhibit wastage rates in the puddle areas that are more typical of those encountered in sea water immersions. Alloys containing large amounts of manganese tend to be

somewhat prone to pitting in marine atmospheres, as are the cobalt- beryllium-coppers. A tendency toward intragranular corrosion has been observed in silicon bronzes and aluminum brass, but its occurrence is somewhat sporadic.

6. Magnesium

A clean, unprotected magnesium alloy surface exposed to indoor or outdoor atmospheres free from salt spray will develop a gray coating that protects the metal from corrosion while causing only negligible losses in mechanical properties. Chlorides, sulfates, and foreign materials that hold moisture on the surface can promote corrosion and pitting of some alloys unless the metal is protected by properly applied coatings. The surface coating that forms on magnesium alloys exposed to the atmosphere gives added protection from further attack. Corrosion of magnesium alloys increases with relative humidity. At 9.5% humidity, neither pure magnesium nor any of its alloys exhibit evidence of major corrosion after 18 months. At 30% humidity, only minor corrosion may occur. At 80% humidity, the magnesium may exhibit considerable corrosion. In marine atmospheres heavily loaded with salt spray, magnesium alloys require protection for prolonged survival.

Indoor Atmosphere The widespread introduction of magnesium into the computer disk drive environment has imposed new standards of stability on the metal because of the need to maintain a clean particle-free atmosphere at the disk/head interface. The corrosion of magnesium alloys in indoor atmospheres increases with relative humidity. At relative humidities up to about 90%, corrosion is very minor, as humidity increases beyond this level, heavier tarnish will develop.

7. Nickel

Nickel and nickel alloys have very good resistance to atmospheric corrosion. Corrosion rates are typically less than 0.0025 mm/yr (0.1 mil/yr), with varying degrees of surface discoloration depending on the alloy

8. Zinc

It is generally accepted that the corrosion rate of zinc is low, it ranges from 0. 13 mm/yr (0.005 mpy) in more moist industrial atmospheres. Zinc is more corrosion resistant than steel in most atmospheres, the exceptions being ventilated indoor atmospheres where the corrosion of both steel and zinc is extremely low and certain highly corrosive industrial atmospheres. For example, in seacoast atmospheres, the corrosion rate of zinc is about 1/25 that of steel. For both steel and zinc, corrosion rates vary significantly among different locations and climates.

Indoor Exposure. Zinc corrodes very little in ordinary indoor atmosphere with moderate relative humidity. In general, a film begins to form at spots where dust particles are present on the surface, the film then develops slowly. This attack may be a function of the percentage of relative humidity at which the particles absorb moisture from the air. However, moisture has little effect on the formation up to 70% relative humidity. The degree of corrosion is related to the relative humidity at and above this point because the zinc corrosion products absorb enough moisture to stimulate the attack to a perceptible rate. Rapid corrosion can occur where the temperature decreases and where visible moisture that condenses on the metal dries slowly. This is related to the ease with which such films maintain a high oxygen content because of the small volume of water and large water/air interface area. Considerably accelerated corrosion can then take place with the formation of a film that is too thick. Atmospheres inside industrial buildings can be corrosive, particularly where heated moisture and gases, such as SO_2, condense near a cool room.

Atmospheric Exposure. Several atmospheric exposure programs have been conducted throughout the world to obtain corrosion rate data for zinc exposed to representative natural atmospheres. These programs have provided quantitative evidence of the excellent resistance of zinc over wide range of atmospheric conditions. Although there is considerable spread in terms of percentage in the corrosion rates observed, the actual corrosion rate rarely exceeded about (0.3 mpy) in average metal loss, even under the more severe conditions. This is well within standards of acceptable corrosion performance. Zinc owes its high degree of resistance to atmospheric corrosion to the formation of insoluble basic carbonate films. Environmental conditions that interfere with the formation of such films may attack zinc quite rapidly. In dry air,

zinc is slowly attacked by atmospheric oxygen. A dense layer of oxide is formed on the surface of the zinc, and a porous outer layer then forms on top of it Although the outer layer breaks away occasionally, the thin under layer remains and protects the metal by restricting its interaction with the oxygen. Under these conditions, which occur in some inland tropical climates, the zinc oxidizes very slowly. The rate of drying is an important factor because of moisture film with higher oxygen concentration promotes corrosion. For normal exposure conditions, the film dry quite rapidly, and only in sheltered areas are drying times slow enough to accelerate the attack of the zinc significantly.

Industrial and Marine Pollution In such circumstances, the corrosivity of the contaminant may be more important the degree of moisture condensation. Sulfur dioxide is one of the most harmful pollutants in the atmosphere, and it plays a major part in the corrosion of steel and zinc. Exposure tests showed that the correlation between sulfur pollution and corrosion is high for copper-bearing steel and for zinc, and these tests demonstrated that the SO_2 concentration in the air is the determining factor for the intensity of the corrosion of these metals. Another series of tests found that even at test sites situated far from industrial towns the corrosion products contained a strong sulfate component derived from atmospheric sulfur compounds. This indicates that the effects of this type of pollution are far reaching.

F.   CORROSIVE AGENTS IN EMAA FORMULATIONS

1.   *Potassium Hydroxide*

Potassium hydroxide, KOH also known as caustic potash, lye, and potassium hydrate, is a white crystalline solid that has a melting point of $360\,^{\circ}$ C ($680\,^{\circ}$ F). It is soluble in water and alcohol. It is a powerful cleansing bath for scouring metals and when used in steel-quenching baths gives a higher quenching rate than water alone without attacking the steel as a salt solution would. Potassium hydroxide is slightly more aggressive in the corrosion of metals and nonmetals than sodium hydroxide.

2.   *Magnesium Hydroxide*

Magnesium hydroxide MgOH, is a white powder that is very slightly soluble in water. It decomposes at 350 ^{0} C (662 ^{0} F). Magnesium hydroxide is formed by the reaction of sodium hydroxide and a soluble magnesium salt solution.

3.   *Potassium Chloride*

Potassium chloride, KCl, also known as potassium muriate and sylvite, is a colorless crystalline solid with a salty taste that melts at 776 ^{0} C (1420 ^{0} F). It is soluble in water, but insoluble in alcohol. Potassium chloride is used in fertilizers, pharmaceuticals, and as a salt substitute.

G.   RESULTS

Results are reported in the following three categories: **corrosion rate** (mils per year), **mass loss percentage** (grams per square meter), and **pitting density** (frequency per square millimeter). Both corrosion rate and mass loss percentage refer to relatively uniform, generalized attack, while pitting density refers to more localized, non-uniform attack.

1.   CORROSION RATES, SFE FORMULATION A

Corrosion rates for coupons exposed to SFE-A formulation (Figure 16) vary from trace corrosion, < 1.0 mpy for monel 400, nickel, and stainless, to much heavier corrosion of carbon steels (1020 and 607), aluminum, and magnesium, (12.0 - 32.0 mpy). Zinc, brass, and copper sustained comparable corrosion rates in the 4.0 - 5.5 mpy range. Mass loss percentages (Figure 17) are comparable to the corrosion rates. Monel 400, nickel, and stainless had trace mass loss percentage, <0.01 %. Zinc, brass, and copper experienced similar losses, ≅ 0.02 %. Steel 607 and aluminum showed mass loss percentages ≅ 0.045%, steel 1020 0.08% and magnesium approaching 0.14%.

CORROSION RATES (mpy)
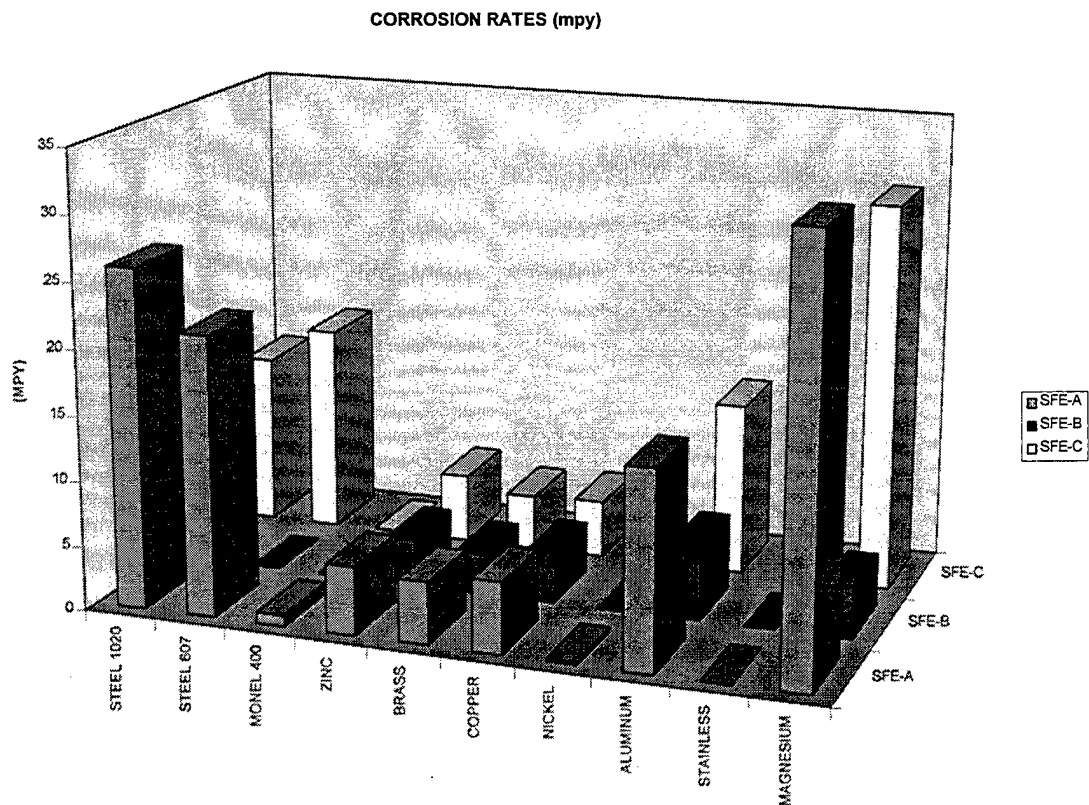

Figure 16   Corrosion rates (mpy), selected metals/metal alloys

MASS LOSS % (G/M2) SFE
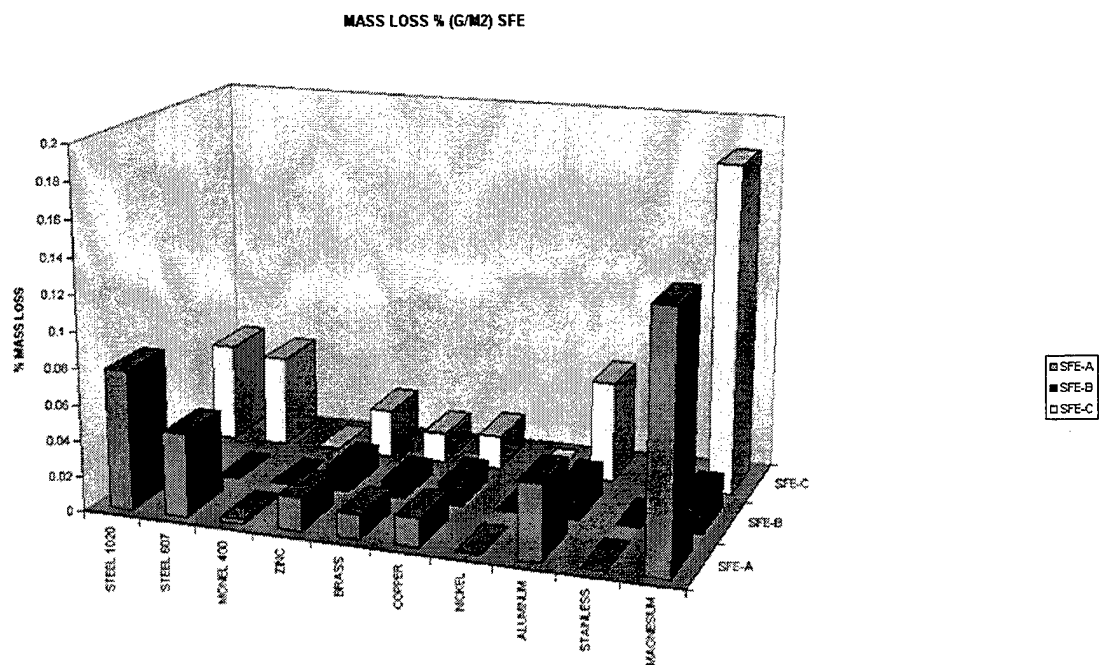
Figure 17   Mass loss percentages (grams/meter^2), selected metals/metal alloys
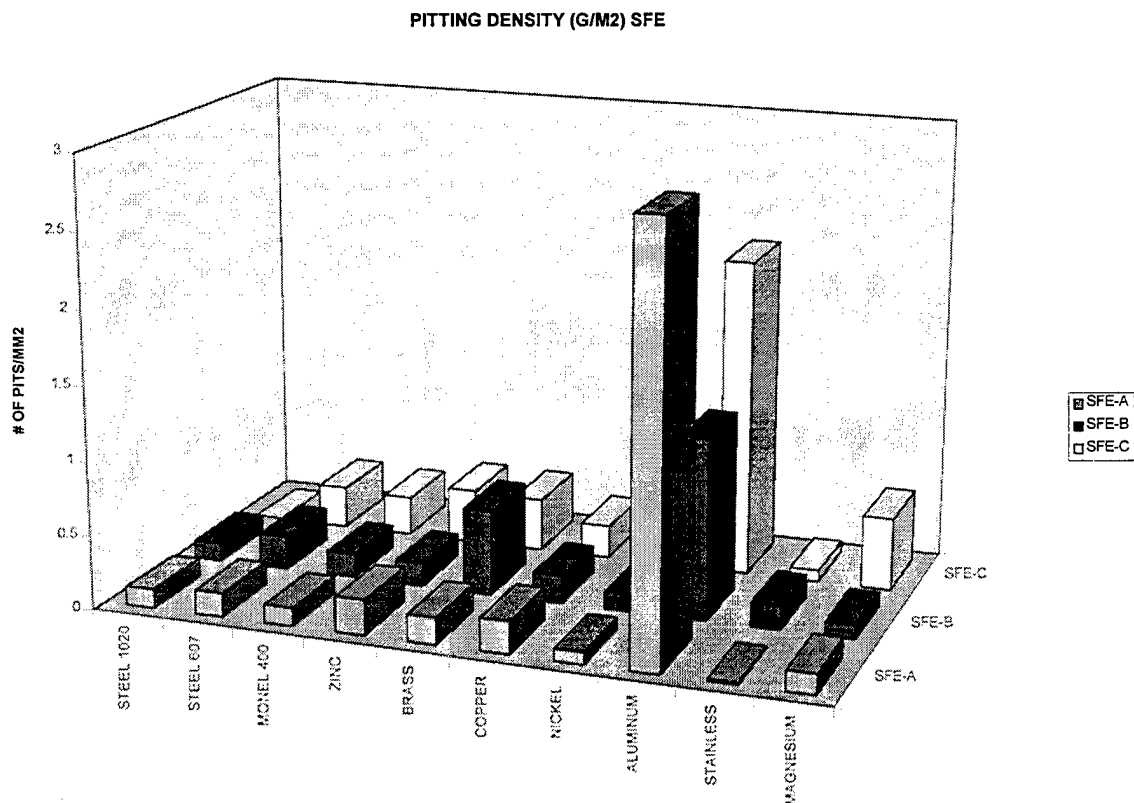
PITTING DENSITY (G/M2) SFE

Figure 18.   Pitting density (frequency/mm²), selected metals/metal alloys

Pitting density was uniform among all sample coupons (Figure 18), < 0.3 frequency per square millimeter, except aluminum which experienced severe localized attack, ≅ 2.7 frequency per square millimeter.

2.   CORROSION RATES, SFE FORMULATION B

Corrosion rates for coupons exposed to SFE-B formulation were far more uniform than those for SFE-A, and fall within acceptable levels for all samples. Monel 400, nickel, steel 607 and 1020, and stainless all had trace corrosion, <1.0 mpy. Zinc, brass, copper, aluminum, and magnesium

had higher, but acceptable rates, 3.0 - 6.0 mpy. Mass loss percentages were also more uniform than SFE-A, with no sample > .024 %.

Eight of the ten samples experienced nominal pitting density. Brass and aluminum experienced some increased frequency of localized attack, although the severity and depth of the pitting was not as great as caused by SFE-A.

3.   CORROSION RATES, SFE FORMULATION C

Corrosion rates for SFE-C were indicative of rates obtained from exposure to SFE-A. Monel 400, nickel, and stainless experienced nominal corrosion rates. Zinc, copper, and brass showed higher, but acceptable rates, and highest rates were again present in steel (607 and 1020), aluminum, and magnesium. Mass loss percentages were also indicative of rates obtained from exposure to SFE-A, as was pitting density.

SECTION V

RECOMMENDATIONS

The various SFE formulations prove effective in fire suppression only when total flooding conditions are achieved. Initial estimates predict total-flooding to occur at 50 grams SFE in aerosol suspension per cubic meter of coverage area. Results from testing indicate that approximately 20 percent of the SFE material does not form aerosol upon combustion, and an additional 15 to 20 percent is lost to deposition in the packed-bed of refractory material. Successful fire suppression was only noted while testing the two-kilogram charge generator. Under ideal conditions 100% efficiency in aerosol generation and unrestricted movement through the refractory material, the 500 gram charge should provide total-flooding for a 10 cubic meter area. Under initial testing conditions, 2000 grams were needed to provide successful fire suppression. This must be considered when determining size, weight, and cost benefits of this type of halon replacement. Additionally, pressure increases due to restrictions placed on rapidly generated aerosol within the generator housing provide a potential for device failure.

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APPENDIX A: CORROSION STUDY: MATERIALS AND METHODS

1.   Equipment and samples required

1) Exposure chamber

2) Temperature/Humidity control chamber

3) Analytical balance scale

4) Microscope with calibrated reticule

5) Non-abrasive detergents

6) SFE 10 gr. charges (SFE-A, SFE-B, SFE-C).

7) Data acquisition hardware or software

8) Sample coupons each of the following metals/metal alloys:

     1) 306-1020 Steel, SAE-1020 Cold-Rolled, 2.5" X 0.25" X 0.125", polished 280 grit.

     2) 306-607 Steel, ASTM A-607, Class 1, Grade 50, 2.5" X 0.25" X 0.125", polished 280 grit.

     3) 306-400 Monel 400, QQ-N-281, Class A, 2.5" X 0.25" X 0.125", polished 280 grit.

     4) 306-Zn Zinc, 99.9% pure, ASTM B-6, Type I, 2.5" X 0.25" X 0.125", polished 280 grit.

     5) 306-260 Brass, CA-260, 2.5" X 0.25" X 0.125", polished 280 grit.

     6) 306-110 Copper, CA-110, QQ-C-576, Annealed, 2.5" X 0.25" X 0.125", polished 280 grit.

     7) 306-200 Nickel 200, ASTM B-162, , 2.5" X 0.25" X 0.125", polished 280 grit.

     8) 306-2024 Aluminum Alloy, QQ-A-250/4 (2024 T-3 Bare), , 2.5" X 0.25" X 0.125", polished 280 grit.

     9) 306-304 Stainless Steel, Type 304, QQ-S-766, Annealed, 2.5" X 0.25" X 0.125", polished 280 grit.

     10) 306-AZ31B Magnesium Alloy, QQ-M-44 (AZ31B H-24), 2.5" X 0.25" X 0.125", polished 280 grit.

9) Plastic film.

10) Data acquisition hardware/software to record temperature and humidity data.

2. Exposure procedure

1) Etch coupons with appropriate identifying numbers and letters, (# for the type of metal, letter for EMAA formulation or control, second # for which sample). Clean all coupons with acetone; dry thoroughly in a dessicator.

2) Using the analytical balance, weigh and record sample coupons.

3) Insert tray into exposure chamber floor.

4) Set SFE charge in opposite corner of chamber.

5) Use a squib to set off charge.

6) Immediately close the chamber.

7) Allow 24 hours exposure.

8) Mark the data stream in the data acquisition hardware/software with the appropriate date and time stamp, and tag it with the name SFEXYY, where X is the formulation used (A,B, or C) and YY are integers which indicate experiment sequence.

9) At the 24 hour mark, remove tray with exposed samples from the exposure chamber, and set tray in the lowest drawer available on the temperature/humidity control chamber and close the chamber.

10) Repeat steps 1 - 9 using SFE-B and then SFE-C.

11) Expose each tray for 30 days.

12) At the end of the 30th day, extract the exposed tray and analyze corrosion according to the method outlined in the following section.

13) Collect all data recorded from the temperature/humidity control chamber.

3. Procedure to estimate total aerosol deposition density

1) Cut four 1" X 1" pieces of a low density plastic film.

2) Weigh each film and record data.

3) Place the films in the four corners at the same plane where samples are to be exposed.

4) Fire the corresponding SFE charge.

5) Wait 24 hours after firing and remove films.

6) Weigh films independently and determine the increase in mass due to aerosol deposition.

7) Compute the aerosol deposition density (increase in mass/area of film)

8) Estimate the total deposition density by computing the average deposition density amongst the four film samples.

4. Procedure to analyze corrosion of exposed samples

1) Using the appropriate solvent, clean the surfaces of the exposed samples using nonabrasive cleaners/solvents.

2) Weigh each sample and record the data.

3) Using a microscope equipped with a calibrated reticule, observe each sample in the microscope at a magnification of 40X and record the number of pits per square mm.

4) Increasing the magnification of the microscope, measure the average diameter of as many pits as possible (a minimum of 20 pits) and record the data.

5) Report corrosivity by computing

   a) % mass loss

   b) Density of pits (pits/mm sq)

   c) Average pit size (mm in diameter)

per unit exposure of each SFE formulation.